FLOTATION OF JAROSITES

by

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FLOTATION OF JAROSITES

INTRODUCTION

The flotation of jarosites is a new problem which promises to be one of growing importance in the field of oxide flotation. In order to properly present the significance of this problem it will be necessary to discuss the rather peculiar position which is occupied by oxidized ores in the mineral industry and also the peculiarities of oxidized ores in their relation to methods of treatment.

In nearly every mining district the upper or weathered portion of the ore deposits contain oxidized minerals. The depth and extent of the oxidized zone depends upon many geological, topographical, and climatical conditions. Oxidation has reached great depths in many of the mining districts of the arid western United States. For instance, in the Tintic district of Utah, the zone of oxidation extends to a depth of at least 1,500 feet, and is known to be much deeper in many mines. Outstanding tonnages of oxidized ores occur especially in the states of Arizona, Utah, and Nevada. These oxidized

zones represent every gradation in value content from those in which the valuable minerals have been almost completely leached, (to be re-deposited in the zone of secondary enrichment) through those in which the valuable minerals are present but converted to oxide forms, to those in which there has been a high concentration of values in the oxidized zone.

The early history of mining has witnessed, in practically every mining district, the exploitation of the high grade oxidized ores near the surface; later the high grade sulphides of secondary enrichment zones; and now by far the majority of mining operations are confined to the more or less complex and lower grade primary sulphides. This change has meant a change from the production of noble metals to the production of base metals containing the noble metals as a by-product. This, in turn, has meant a transition in metallurgical practice. The first high grade oxidized ores were direct smelting. Later, in the transition period, the mixed ores of the partly oxidized ore zone had to be treated instead of the direct smelting and free milling ores of the past or the deeper occurring sulphide ores that were to be subsequently smelted. At this period the flotation process had not been discovered and processes such as gravity concentration, leaching, and volatilization were developed.

Flotation came in after the bulk of mining operations had turned to the primary and complex sulphides and in its phenomenal development the most attention has naturally been paid to sulphide problems.

Flotation of oxidized ores is still in its infancy. Yet it

has reached a status where the economic marginal point of ores has been lowered in general from that obtaining by the processes above mentioned. This makes available increased tonnages of oxidized ores previously below economic margin. Also, flotation of the sulphides has imposed a change of subsequent metallurgical treatment which in itself affects the demand for oxidized ores--that is, the need for silicious material to flux the highly pyritiferous concentrates now made by flotation. Again while flotation determines the amount of iron left in the sulphide concentrate it also decreases the silica in the oxidized ores. It is evident that a balance is in continual adjustment between the oxidized ore required for smelting and the economic margin enforced on such an ore by its contained values and transportation costs. Although there is this economic justification for the direct smelting of a large portion of the oxidized ores, it must not be overlooked that large tonnages of the ores considered as sulphides contain varying amounts of oxidized minerals which are usually a cause of much loss in concentration. In addition should be considered those oxidized ores which, because of their grade and location can not withstand freight charges, yet could be profitably handled by concentrating.

All of these factors, plus economic conditions, aside from those resulting from improved technical developments, have contributed to the present status of oxidized ores. Large tonnages of low grade oxidized ores encountered in earlier mining operations have been left because there were then no processes by which they could be profitably treated.

Low grade oxidized ores and mixed oxidized and sulphide ores have been left underground by some of the newer mines which have passed through the oxide zone to develop a more profitable sulphide zone, or have mined only the higher grade portions of the oxidized bodies. Because there has been this tendency to avoid this type of ore it is hard to estimate the amount of this low grade material available. Also, where such ores have in the past been treated by preliminary concentration there are dumps of slime tailings containing appreciable oxide mineral values due to the well known tendency of oxide minerals (particularly lead carbonate) to "slime". As already mentioned there is, in addition to these quantities, the varying amounts of oxidized and semi-oxidized mineral present in much of the sulphide ore treated.

That there is a realization of this situation is reflected in the fact that each year there is a slight increase in tonnage of oxidized ore milled.[1] Oxidized copper ores have, in general, lent themselves quite readily to the flotation process and considerable tonnages of such ores are being treated, principally in Arizona. Since this paper deals with a mineral of lead and silver, further discussion will be narrowed to oxidized lead-silver ores. Several of the most important mining companies of the Western United States and Mexico are now operating flotation plants for treating oxidized ores. A·W. Hahn (in paper quoted on page 4), and Thomas Varley,[2] have

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[1] Hahn, A.W. Flotation of Oxidized Lead-Silver Ores. Tech. Publication No. 10, A.I.M.M.E., 1927

[2] Varley, Thomas. The Flotation of Oxidized Ores. Reports of Investigations, Serial No. 2811, U.S.Bur. of Mines, 1927.

given resumés of these plants. According to these authors, the approximate total tonnage for these mills is about 1400 tons per day (these figures vary considerably from year to year according to the number of plants in operation). To these are now added the Chief Consolidated Mining Company's mill at Eureka, Utah, and the new oxide mill of the International Smelting Company which began operation in 1929, at Tooele, Utah.

The outstanding feature in the flotation of oxidized and semi-oxidized ores is that in general, losses are high as compared with losses in sulphide flotation. There is a combination of reasons for this, the first of which is the nature of oxidized ore itself. Oxidized ores may be considered those whose components, usually originally present as sulphides, have undergone a change in chemical composition by natural weathering.[3] Oxidized ores are perhaps more commonly referred to as carbonates. Speaking of oxidized lead ores, usually lead carbonate is present in the greatest abundance by far, lead sulphate is common, and oxides and other rarer forms, common but in much less quantity. The transition to the oxide forms involves decomposition of minerals and changes in chemical components dpending on many conditions and agencies. Thus there may be all gradations in transformation of minerals from true sulphides to true oxides. The number of different minerals which may occur in

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[3] Varley, Thomas. loc. cit. p-3

oxidized ore deposits is surprising. (See Table 1).

In examining this table it is well to bear in mind that the ore deposits in which these minerals may develop are usually high in iron. This is probably originally present as the sulphide and in the many changes in decomposition of the sulphides and formation of sulphates, carbonates, hydroxides, and oxides, it is conceivable how a definite mineral of the composition of jarosite may commonly develop. Also, it is conceivable how such a mineral may be occluded or massed by the iron compounds in the various stages of transition and thus fail to be recognized as a mineral carrying lead or silver values. To substantiate this view the following is briefly abstracted from the Data of Geochemistry:[5]

> "In the first place, the surface waters, charged with oxygen and carbonic acid, attack the outcrop of ores, oxidizing them more or less completely to sulphates. Sulphuric acid or acid salts are formed at the same time, which assist in the decomposition of the adjacent rocks. That decomposition is more than ordinarily extensive in the vicinity of metalliferous veins, and the rocks therefore acquire a higher degree of permeability to the percolating waters."[6]

> "Ferrous sulphate first oxidizes, yielding ferric hydroxide and insoluble basic salts."[7]

> "R. Warington found that hydroxides of iron and aluminum, particularly the former, were especially active as absorbents, and most so in the presence of calcium carbonate..... Warington's experiments point directly to an absorption by colloids, namely, the colloidal hydroxides of iron and aluminum."[8]

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[5] Clarke, F.W. The Data of Geochemistry. Bull. No. 770 U.S.Geological Survey, 1908

[6] Ibid-p.652

[7] Ibid-p.536

[8] Ibid-p.504

TABLE I

A LIST OF THE BETTER KNOWN OXIDIZED LEAD MINERALS[4]

Lead Compound	Formula	Specific Gravity
Lead carbonate (cerussite)	$PbCO_3$	6.6
Basic lead carbonate	$2PbCO_3.Pb(OH)_2$	6.14
Anglesite	$PbSO_4$	6.2
Massicot (lead monoxide)	PbO	8.0
Plattnerite (lead dioxide)	PbO_2	9.37
Minium	Pb_3O_4	9.1
Barysilite	$3PbO.2SiO_2$	6.72
Vanadinite	$(PbCl)Pb_4V_3O_{12}$	6.86
Descloizite	$R_2V_2O_8.R(OH)_2$ (empirical)	6.0
Wulfenite (lead Molybdate)	$PbMoO_4$	6.7
Pyromorphite	$3Pb_3P_2O_8.PbCl_2$	6.8
Plumbojarosite	$PbO.3Fe_2O_3.4SO_3.6H_2O$	3.63
Crocoite	$PbCrO_4$	6.3
Phosgenite (chlorocarbonate)	$PbCO_3.PbCl_2$	6.13
Ganomolite	$3PbO.2(Ca,Mn)O.3SiO_2$	5.74
Melanotekite	$2PbO.Fe_2O_3.2SiO_2$	5.73
Leadhillite	$4PbO.SO_3.2CO_2.H_2O$	6.5
Lenarkite	Pb_2SO_5	6.92
Caledonite	$(Pb,Cu)SO_4.(Pb,Cu)(OH)_3$	----
Linarite	$(Pb,Cu)SO_4.(Pb,Cu)(OH)_2$	5.4
Corkite (hydrous phosphate) and sulphate of Pb and Fe)	Doubtful	4.2
Beaverite	$CuO.PbO.Fe_2O_3.2SO_3.4H_2O$	4.36
Dechenite	PbV_2O_6	----

[4] International Critical Tables. Mcgraw Hill, New York, 1926, vol. 1, p - 160.

> "the precipitated hydroxides of iron vary much in character and appearance, and their exact chemical nature, despite the plausible formulae assigned to some of the minerals, is by no means clear. In color they range from yellow through various shades of brown and red, and in texture they differ as widely." [9]

The silver minerals are as numerous as those of lead, but unlike lead minerals, the only one true oxide silver mineral occurring in nature is argentojarosite.[10] A very good idea of the ways in which silver may appear in oxidized ores may be had by quoting from a recent paper by A.W. Hahn[11] on ores of the type under discussion:

> "These chemical and mineralogical determinations showed the silver to be present as native silver, argento-jarosite, argentite, and combined or inherent in the tetrahedrite, chalcopyrite, galena, pyrite, cerussite, englesite, mimetite, malachite, iron oxide and occluded in the quartz. Careful microscopic work would probably show other forms in small amounts, such as the halide silver minerals and some of the antimony sulphide combinations."

As in the case of the lead minerals, the significance of the plentiful presence of iron, during the various alteration stages is obvious. The following abstract is given,(although it is in reference to sulphide enrichment) for the possibility it discloses on occlusion of silver values and argentojarosite formation.[12]

> "Sulphide enrichment of a primary silver deposit can be brought about by reactions of silver or silver sulphide with the sulphides of iron and their products of oxidation. When the latter is pyrite (continued on page 9)

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[9] Clarke,F.W. loc. cit. p-537
[10] Clarke,F.W. loc. cit. p-670
[11] Hahn,A.W. Silver Bearingiminerals of Some Ores from the Tintic Mining District. Tech. Pub. No. 202, Class B,A.I.M.M.E. 1929

> the Ag_2S is converted into Ag_2SO_4 by the H_2SO_4 and $Fe_2(SO_4)_3$ produced. Except in the case of galena the solvent action does not seem to be proportional to the conc. of the $Fe_2(SO_4)_3$. Equilibrium in Ag bearing solns between sulphates is such that the reduction of Fe''' to Fe'' will ppt. metallic Ag. Ppt'd S may convert it into Ag_2S again."

Here it seems very possible, at the stage where silver sulphide is converted into silver sulphate by sulphuric acid and iron sulphate, that argentojarosite, the basic iron sulphate of silver, might be formed.

C. A. Shempp[13] has advanced the following thory for the origin of argento jarosite:

> ".....that silver sulphide has been dissolved in a solution of basic ferric sulphate, and the mineral was crystallized from the solution. Its occurrence is such as to confirm the opinion that it is secondary."

With the increasing valuable application of the microscope to study of ores and the products of metallurgical processes has come an increasing realization that not enough attention has been paid to the occurrence of supposedly uncommon minerals in oxidized ores. It is fundamental knowledge that the forms in which the mineral constituents are present in an ore have an all important bearing on the method employed for their recovery. particularly is this true in flotation. In addition to the various forms in which the valuable minerals may occur in an oxidized ore deposit must be considered the forms of the gangue minerals. It has been stated that most of the ore deposits of the minerals under consideration

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[12] Chemical abstracts. Vol. VII, page 1456.
[13] Shempp, C.A. Argento-jarosite; a New Silver Mineral. Amer. Jour. of Science, 5th series, vol.6, 1923. pp.73-75

contain much iron and the quantity of the oxidized iron minerals and the properties of minerals developed in this presence can be expected to vary greatly. The weathering of gangue minerals along with the valuable minerals causes complications such as the development of abundant slime material (for instance by kaolinization and sericitization of feldspar); varying degrees of surface alteration; filming and occulsion of valuable minerals by gangue minerals (for instance coating by iron oxides); and development of soluble salts in the flotation circuit. Mechanical difficulties, high losses in the tailing, dilution of the concentrate, and high consumption of reagents are among the difficulties arising from the above conditions.

The importance of recognizing these conditions cannot be over emphasized if successful treatment of oxidized ores by flotation is to be accomplished. There is abundant evidence that in practice many of the above enumerated conditions common to oxidized ores have, perhaps, not been given due consideration. One reason has been the tendency to follow developments in sulphide flotation practice without particular study of the flotative properties of oxide minerals or their peculiar gangue relationships. Study of pure minerals has added greatly to fundamental data on sulphide minerals[14] and hence the interpretation of flotation phenomena and the function of reagents.[15] However, many of the minerals and

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[14] Tech. Paper 1. 2, and 3. Utah Eng. Expt. Sta. 1928

[15] Gaudin, A.M. Flotation Mechanism. A Discussion of the Functions of Flotation Reagents. Tech. Paper No.4. A.I.M.M.E. Aug.1927

problems of the oxidized ores present totally new fields of investigation. Microscopical investigation coupled with chemical determinations and with actual flotation work on pure and mixed minerals affords an excellent avenue for results.

Investigations in line with this method of attack have been carried out on oxidized silver-lead ores by R·E.Head and Virgil Miller.[16] The following is quoted from their Reports on Investigations:

> "Recent investigations at the Intermountain Experiment Station of the Bureau of Mines at Salt Lake City indicate that metallurgists have given little attention to the occurrence of jarosite minerals in the oxidized ores of lead and silver. These investigations have also emphasized the importance of knowing when those minerals occur in the oxidized ores, especially those containing lead, silver, and gold, as their presence is a material factor in selecting a suitable kind of treatment. Since the question of the occurrence of jarosites in oxidized ores has come to the attention of the Bureau of Mines investigation has indicated that plumbojarosite and argentojarosite are quite common. These two materials appear to be widely distributed and probably escaped recognition by persons who had attempted to treat certainooxidized lead ores in which the jarosite minerals represented a material portion of the metal content....
>
> "There are undoubtedly considerable tonnages of oxidized lead ores in the various mining regions of this and other countries, and it is quite probably that in some of these ores the metal content of lead and silver may be largely anglesite (lead sulphate) and cerussite (lead carbonate) but that they will also contain cerargyrite (silver chloride), argentojarosite, and plumbojarosite. The treatment of such ores therefore presents an important problem since a product is discarded from ores on which the recoveries of lead and silver have been below 70 per cent which is

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[16] Head,R.E. and Miller,Virgil. The Occurrence of Jarosite Minerals in Oxidized Lead Ores as a Factor in Metal Losses. Reports of Investigations,Serial 2870, U.S. Bur. of Mines, April 1928.

probably too low-grade to warrant after treatment by any process now in use.....

"....Where tailings have been discarded which contain 1 or 2 ounces in silver and several per cent in lead, existing chiefly as jarosites there is some question as to whether they would stand the expense of any metallurgical treatmnet now known. Such material therefore, represents an economic loss.

".....The presence of the jarosite minerals in oxidized ores has probably been overlooked by the metallurgist and mill man, who have assumed that the brown color in many ores was due to the presence of the hydrous iron oxides."

In addition to demonstrating conclusively that the occurrence of jarosite minerals in oxidized lead ores is a factor in metal losses the paper gives excellent material on the character and occurrence of jarosite minerals and also the results of some preliminary investigations in the line of leaching, roasting, and sulphidizing. Main features of the results of these investigations, stated very briefly,are as follows:

Careful examination should be made of tailings from milling of oxidized lead ores on which lead and silver recoveries are low, to determine whether the losses are due to the presence of jarosites. Recovery of jarosite minerals by gravity concentration is practically impossible because of their low specific gravity, small size, and micaceous character. The jarosite minerals do not sulphidize when using concentrations of reagents and time periods ordinarily employed in practice. Extraction of values from jarosite minerals by leaching does not appear to be economically feasible. Results of volatization

experiments on several jarosite ores suggest further consideration of this method.

Since this is practically the only work that has been previously done from the metallurgical standpoint, on jarosites, it represents the status of the problem at the beginning of the present investigation.

THE JAROSITE MINERALS

Description, Characteristics, and Occurrence.

Because of the lack of knowledge which is indicated on the subject of jarosites, a brief description and discussion of their characteristics and occurrence may be of general value and will throw light on some features of the following work. There are five species in the jarosite group to which the following general formula has been given: $RO.3Fe_2O_3.4SO_3.6H_2O.$, When R is substituted by K_2, Na_2, $(NH_4)_2$, Pb, or Ag_2, the formulae are given respectively, for jarosite, natrojarosite, ammoniojarosite, plumbojarosite, and argentojarosite. The mineral is thus considered a basic iron sulphate of each of the above elements. Sometimes the formula is given as $R{\cdot}Fe_6(OH)_{12}(SO_4)_4$. Obviously plumbojarosite and argentojarosite are the members of this group with which the metallurgist will be principally concerned and this work has been limited to these two. Folowing are analyses given for plumbojarosite by Hillebrand and Butler,[17] and for argentojarosite by Shempp and Schaller:[18]

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[17] Schaller, W.T. Mineralogical Notes, Series 2; U. S. Geological Survey Bull. 509, 1912. 115pp.

[18] Shempp, C.A. Argentojarosite--A New Silver Mineral. Amer. Jour. of Science, 5th series, vol.6, 1923. p.73-75

Plumbojarosite	Per cent.	Argentojarosite	Per cent.
Fe_2O_3	42.38	Ag_2O	18
Pbo	19.74	Fe_2O_3	43
SO_3	28.83	SO_3	28
H_2O	9.55	H_2O	10

All of these minerals crystallize in the hexagonal system. They usually develop with basal cleavage and hence appear in hexagonal outline in thin, flat micaceous greins. The crystals usually appear in platy tabular groups and close examination indicates that they are possibly held in these clumps by a thin cementing of iron oxide.

The individual crystals are very minute. Microscopical measurement of specimens from several localities indicate a size range from 0.100 mm to 0.003 mm, with perhaps an average range for plumbojarosite and argentojarosite of 0.080 mm to .010 mm.

The color is similar for all and varies from lemon yellow and golden yellow to brown, which accounts for their ready confusion with iron oxides.

They are all soft, the hardness being considered as between 2.5 and 3.5 Mohr scale.

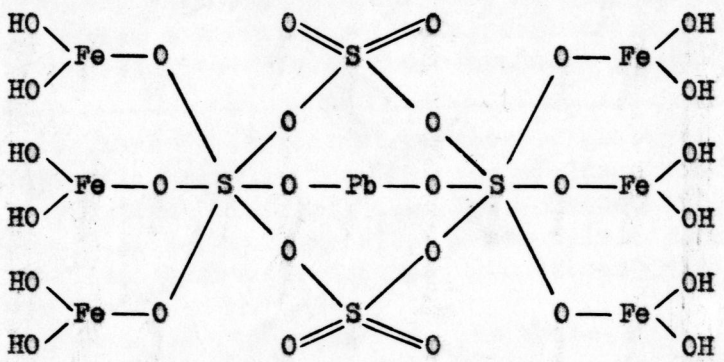
Specific gravities range from 3.15 for jarosite, (potassium), to 3.665 for plumbojarosite. This is of great importance in considering a method of concentration, for it can be seen by reference to Table 1,

page 7, that this specific gravity is considerably lower than that of any of the other oxide lead minerals listed.

Occurrence of jarosite minerals has been definitely established in many of the western mining districts. A list of these districts together with some notes and analyses is given by Head and Miller.[19]

Following are abstracts from these and additional sources which will serve to show to some extent the distribution of jarosite minerals:

> Members of the U. S. Geological Survey[20] report the first recognition of plumbojarosite at Cook's Peak, New Mexico. This discovery of plumbojarosite indicates the isomorphism of potassium, sodium and lead. It is believed that they are solfateric products, formed under combined action of heat and pressure. They are formed, like many precipitates, as fine crystalline powders because difficultly soluble. The authors, from their study, give the following as perhaps the most satisfactory way of writing the formula:



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[19] Shannon,Earl V. Ammoniojarosite, a New Mineral of the Jarosite Group from Utah. Amer. Mineralogist, vol. 12, No. 12. Dec. 1927. pp.424-426
Hillebrand,W.F. and Penfield,S.L. Some Additions to the Alunite - jarosite Group of Minerals. U.S.Geological Survey Bull.262 1905. pp.32-41
Schaller,W.T. Mineralogical Notes, series 2; U.S.Geological Survey Bull. 509, 1912, 115 pp.

Butler states:[21]

"Long previous to the recognition of the mineral in 1902, Dr. Hillebrand had made an analysis of a ferric sulphate from Leadville, Colorado, that from its close correspondence to the analyses of plumbojarosite, gives a strong reason to suspect that it was that mineral rather than a mixture of minerals as was then thought....."

"In 1911 the writer and Dr. W. T. Schaller described the occurrence of plumbojarosite from six localities in Beaver County, Utah..... Since studying the occurrence of plumbojarosite in Beaver County, the writer has visited numerous other camps in Utah and has found that plumbojarosite is of wide spread occurrence, being collected from no lees than 18 localities, including those mentioned above. In some of these occurrences it is present in considerable quantity, and, while not of high grade, is a commercial ore mineral."

Quoting from Knope:[22]

"This Au-Pt-Pd deposit at the Boss mine, Clark Co., Nevada, is not closely similar to any other deposit carrying Pt metals heretofore described. The ore shoots, occurring in dolomite, consist of fine grained, siliceous ore carrying Bi-bearing plumbojarosite, a greenish yellow mineral of smooth unctuous feel, seen under the microscope to consist of

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[19] Shannon,Earl V. (assistant curator of geology, U.S. National Museum). The Minerals of Idaho. Smithsonian Institution Bull. 131, U.S. Nat. Museum, 1926.
Shempp,C.A. Argentojarosite; a New Silver Mineral. American Journal of Science, 5th series. vol. 6,1923. pp. 73-75

[20] Hillebrand,W.F. and Penfield. (as above)

[21] Butler,B.S. Econ. Geol. vol. 8, 1913. pp. 311-322.

[22] Chemical Abstracts. Vol. IX, p.2633, 1915. (Adolph Knope. U.S. Geological Survey).

perfect hexagonal plates averaging 0.01 mm. in diam. It carries Bi_2O_3 6.34, Au 0.79, Pt 0.05, and Pd 0.22 %.... The mineralogical comp. of the material was found to be 80.58% plumbojarosite and 20.01% beaverite..... Vegasite, also present, behaves qualitatively like plumbojarosite and is probably best interpreted as a basic sulphate, $Pb\left[Fe(OH)_2\right]_4(SO_4)_3$ + 10% colloidal (Fe,Al) OH_3."

To quote from Butler's, ORE DEPOSITS OF UTAH:[23] "Plumbojarosite is rather common as an oxidation product in the oxidized lead ores of Utah. It has been recognized in the Star, San Francisco, Tutsagubet, Tintic,Ophir, Stockton, Lucin, Fish Springs, Clifton, Big Cottonwood, Little Cottonwood, and American Fork districts. In several mines it is a valuable ore mineral."

Referring to the Gold Hill mine, Butler says:[24] "Oxidation of the ore has been very complete..... The lead ore is of particular interest in that much of the lead is present as the hydrous ferric lead sulphate plumbojarosite."

Clarke states:[25] "Plumbojarosite, a highly hydrated sulphate of lead and iron is abundant in some mines in Utah."

C.A. Shempp,[26] who was the first to discover argentojarosite describes the discovery which was made in the Tintic Standard Mine:

".....on August 19, 1922, while drifting on the 900-foot level of the Tintic Standard mine, a yellow streak was found which carried very high silver values, assays on the ores running 300 to 1200 oz. of silver per ton. The mineral itself

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[23] Butler,B.S. The Ore Deposits of Utah. U. S. Geological Survey Professional Paper 111, 1920. p. 116

[24] Ibid--p.481

[25] Clarke, F.W. The Data of Geochemistry. U.S. Geological Survey, Bull. 770, 1924. p. 697

[26] Shempp, C.A. loc. cit. (See reference p-11).

> was so finely disseminated through the rocks, as to not be readily recognized. In February, 1923, this streak opened up into a large body of ore from which specimens of sufficient size for analysis were obtained....."
>
> "In the particular place where found, the silver mineral is of common occurrence. It has been the principal silver-bearing mineral in a relatively large tonnage of ore. In the months of February, March and April, the aggregate of the shipments amounted to 200,000 ounces of silver. One carload of ore containing 64 tons assayed 1,018 ounces per ton."

Head and Miller[27] investigated an oxidized lead ore from Tombstone, Arizona, which had been sent in with the hope that something of its characteristics could be learned so that it could be treated without undue metal loss, a difficulty met with in every process so far tried. The investigators discovered that the major portion of the lead was present as plumbojarosite; and also that probably the major portion of the silver was present as argentojarosite. Gold bearing jarosite ores were also studied in these investigations. These ores came from Beatty, Nevada, and Marysvale, Utah. The experimental work indicated that fine gold particles were coated with jarosite particles.

In studying ores from the Alta district, Utah,[28] it was concluded that a considerable portion of the ore was not cerussite and that this portion was principally plumbojarosite.

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[27] Head and Miller. loc. cit. (See reference p-13)

[28] Miller and Head. Roasting of Lead-Carbonate Ores Preliminary to Gravity Concentration. U.S. Bureau of Mines, Tech. Paper 413, 1929.

METHOD OF PROCEDURE

Previous work at this Station[29] has demonstrated the advantage in flotation research of working on pure minerals and synthetic mixture of pure minerals and thus arriving at fundamentals of the process which can then be applied to ores rather than working out the peculiar idiosyncrasies of a large number of ores. Since the flotation of jarosites is a new problem it seemed most logical that it should be attacked in this manner and fundamental data first obtained on the pure mineral.

However, there are certain factors to be considered in the present work which have led to some deviation from the scheme of procedure outlined in the above referred to work. In the first place, although the jarosite minerals have been found widely distributed, they usually occur disseminated in ores and it is difficult to obtain a sufficient quantity of any high degree of purity for experimental work. Also, no quantity of a high degree of purity could be obtained by gravity concentrating a sample containing the mineral, because of its low specific gravity and other peculiarities

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[29]Gates and Jacobsen. Some Flotation Fundamentals and their Practical Application. University of Utah Eng. Exp. Station, Bull.16, 1925

which have been discussed in the introduction. Because of the above fact, and because the control of metal loss due to jarosites is a subject of immediate importance, it was therefore thought desirous to carry the investigation into conditions employing actual associations of the mineral as early as knowledge of fundamental facts on the pure mineral warranted such procedure.

These several factors, peculiar to this problem, combined with the fact that it was entirely new, made it desirous to eliminate all possible non-essentials and consider only the most important phases. This has led to some modification of the usual procedure. In the main, this modification has been to cut down the number of tests for any particular reagent or combination of reagents to a minimum which will indicate in general what is to be expected rather than running detailed series of tests for each case. In doing this, full cognizance has been taken of the fact that reliable results for which explanations may be given can only be obtained by dealing with one variable at a time and holding all other conditions constant. However, there are several cases in which only one or two tests have been made. In these cases there is, of course, not enough data for plotting curves, but they do have a value in indicating a certain possibility or trend.

Aside from these factors there is the interpretation of flotation results--the quantity of jarosite mineral present in the heads and various flotation products. This last presents a somewhat different problem because there is as yet no method of differential

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[30] Miller and Head. loc. cit. p-4

analysis for argentojarosite. The very close similarity of plumbojarosite and argentojarosite eliminates microscopical analysis, because it would not give enough certainty for reliable determination of flotation results. Thus a chemical phase is introduced, i.e., a further preliminary investigation of the chemical nature and physical structure of jarosite should be made, and a differential method of analysis for argentojarosite developed.

A method of differential analysis for plumbojarosite has been worked out by Virgil Miller.[30] Check results are obtainable by this method when certain precautions, to be mentioned later, are observed.

Various samples of jarosite-bearing ores were first obtained. These were examined microscopically and analysed chemically for plumbojarosite by the Miller method. This method is as follows:

> "Place a 1 to 5 gram sample in a 250-cc. beaker and add 50 cc. of a 10 per cent solution of sodium hydroxide, heat to boiling point, let stand for about one-half hour, and filter through an asbestos mat on a Gooch crucible. The filtrate contains the lead from the oxides, carbonates, and sulphates, and the residue contains lead sulphide and plumbojarosite. To separate the sulphide from the plumbojarosite, treat the residue with 25 cc. of a saturated brine solution containing 50 grams per liter of ferric chloride salt. Let stand at room temperature overnight filter and wash thoroughly with hot water, then determine the amount of lead in the filtrate. The filtrate contains the lead originally present as the sulphide. The residue contains the lead as plumbojarosite and is determined by the standard method for lead analysis."

A sample of sufficient quantity for fundamental flotation tests and of high purity was secured from Mexico.[31] This was prepared in definite size ranges. The small size in which jarosite crystals universally occur, and the ready tendency of the minerals to crush to finer

[31] Cia. Minera de Plomo S.A. Villa Felix U Gomez, Chihuahua, Mex.

sizes was given consideration in the flotation work. Preliminary tests were made to determine the effect of the frother and the effect of the degreee of acidity and alkalinity of the circuit on recovery.

This was followed by sulphidizing tests. Recovery by sulphidizing, if possible, would be most desirable because nearly all flotation practice with oxidized lead-silver ores depends on sulphidizing. Sodium sulphide is by far the most widely used sulphidizing reagent. The fact that jarosite minerals escape so readily in the tailing, plus the evidence of non-sulphidizing obtained by Head and Miller[32], indicates that the jarosite minerals behave differently towards sulphidizing action (at least with sodium sulphide as the reagent) than do the other oxide lead-silver minerals. Hence, this is an important phase of the problem.

Work was done with the fatty acids as collectors because of the success which has been indicated by their use in the field of oxide flotation.[32,34] Some of the higher xanthates were used for the same reason.

The work has been concentrated on collecting agents rather than on depressants because of the suppoded natural tendency of jarosite to be unresponsive to collectors. Also, on the ores in which jarosites occur, usually only one or two concentration products are made and it would be desirable to float plumbojarosite and argentojarosite with these products. There would be,probably, no cases where there would be any advantage in attempting to float the jarosites as a separate product.

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[32]Head and Miller. loc. cit.

[33]Gaudin,Hansen,Glover. Flotation Fundamentals,Part I. Tech. Paper No.1, Utah Eng. Exp. Sta., 1928, pp-56-101. (In this work varying degrees of success have been obtained with fatty acids and soaps on:quartz; feldspar; calcite; cerussite; cassiterite; hematite; siderite; magnesite; rhodochrosite).

The work done on separation tests was necessarily brief because it was soon discovered that a thorough treatment of this phase would require a considerably extended program as well as a more complete s solution of some of the problems encountered in the preliminary work on the pure minerals. Consequently this phase was treated in a manner to develop generalizations which it was hoped would indicate the most important avenues for further investigation.

Chemical analyses, both quantitative and qualitative, and the microscope have been used in conjunction with flotation tests wherever such means might aid in developing a theory or eliminating questionable factors in an explanation. The work has been conducted not only with the aim of developing economically practical results as regard the specific problem, but also with an endeavor to contribute to fundamental understanding in flotation of oxide minerals and general flotation theory.

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[34] Iverson,H.G. Separation of Feldspar, Mica and Quartz; work to be published as a technical paper of the Dept. of Mining and Metallurgical Research, University of Utah, in 1930--personal communication.

THE MINERALS USED AND THEIR PREPARATION

The jarosite ores used in this work were obtained through the courtesies of the Tintic Standard Mining Co., Dividend, Utah; Cia. Minera de Plomo S.A., Villa Felix U Gomez, Chihushua, Mexico; and the Silver-Lead Mines Co., La Plata, Utah, to which companies grateful acknowledgment is made.

Ore ore from the Tintic Standard Mine was obtained directly from the loading bins and was representative of oxidized ore being shipped for concentration. A sample of about 600 pounds was secured. This was carefully cut down to a representative sample weighing about 25 pounds and this prepared for flotation feed by crushing through a laboratory jaw crusher, set of rolls, and disc pulverizer to -65 mesh. A portion of this size, undeslimed, was reserved. (See Table II for analyses). The remainder was deslimed by sedimentation, the settling rate being based on time necessary to remove -560 mesh plumbojarosite. It is full recognized that with the many minerals present this procedure would not give accurate sizing, but it is the nearest approach that can be made to obtaining feed which will correspond to that prepared from the pure mineral, so that comparisons may be drawn. Both the sands and slimes were saved.

The ore obtained from Chihuahua, Mexico, was used as the pure mineral for the major part of the work. Under the microscope it appeared to be wholly made up of typical jarosite crystals. These crystals appeared uncontaminated in anyway with other minerals.

The only other mineral present in detectable quantity was a small amount of gypsum. There was no intra-association of the gypsum and plumbojarosite crystals and the two were entirely freed from each other in the sizes employed for flotation. An attempt to remove the gypsum by tabling was made but was unsuccessful because of the little difference in the specific gravities of the two plus the fact that crushing caused the soft gypsum to flatten out into thin flakes which acted about the same as the thin jarosite crystals. Later, in connection with the work on fatty acids, a method for complete removal of the gypsum was devised. This will be discussed under the section on fatty acids.

The fact that repeated analyses (see Table II) showed that the lead was not all present as plumbojarosite, but some as sulphide and as carbonate lead,[35] although the microscope in preliminary examination revealed nothing but very pure jarosite, led to closer examination. Very carefully preformed analyses by the Miller method in which each possible source of error was investigated led to the same result in each case. To test for the presence of some previously unobserved complex minerals, which might have been causing a constant error in the above analyses, a special analytical procedure[36] was used and the residue again run as before. The results checked with those formerly

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[35] Analyses by the Miller method for plumbojarosite; both the molybdate and chromate methods were used for final determination of each portion and independent analyses made by both Mr. Cowles (U.S. Bur. of Mines analyst) and the author.

[36] Procedure obtained through courtesy of Mr. Virgil Miller, who obtained the method in personal communication with an Arizona Company. The method gives quantitative determination for lead present as chloride, sulphate, carbonates, molybdates, tungstates, vanadates, and complex replacement minerals, as well as lead sulphide.

obtained. Careful microscopic examination was again made in which a sample of the pure plumbojarosite crystals was repeatedly ground panned, and examined.[37] When grinding had reached an exceedingly fine stage a streak of sulphide lead was developed. This gave final confirmation of the chemical analyses. These discoveries led to the conclusion that the portion of lead apparently present as sulphide and carbonate was locked up as such in the interior of the jarosite crystals in some manner not as yet exactly understood. (See formula on page 16).

However, it is believed that the jarosite crystals of this ore may be correctly considered as possessing at least, a surface which is purely jarosite mineral. This belief is backed by the fact that the mineral shows unquestionably under the microscope nothing but the very typical jarosite crystals of the correct form, color, luster, and every physical property belonging to jarosite, but no evidence of the other two minerals. To this is added the fact that the mineral is quite insoluble and unresponsive to flotation reagents that should exhibit different results if either the sulphide or carbonate forms were evident. Also, it seems significant that though in sizing the mineral was repeatedly agitated and washed,[38] each size range, including the coarsest used and the -560 mesh slime, gave exactly the same percentage of each lead compound as the head sample. In addition there was no change in composition in flotation concentrate and flotation tailing.

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[37] This work was carried out in final form by Mr. Head, Microscopist, U.S.Bureau of Mines.

Hence, this mineral was used in flotation tests as a pure mineral and recovery in any test was considered as represented by the total weight recovered. The mineral was prepared for flotation feed by hand grinding in a large porcelain mortor to -65 mesh, desliming -560 mesh material, and screen sizing into lots of -65 + 100, -100 + 150, -150 + 200, and -200 + 560 mesh sizes. The porcelain mortor was used for grinding rather than the usual gringing equipment to avoid contamination with metallic iron; it has been found that it is impossible to grind even a soft mineral through the disc pulverizer without smearing the particles with thin flakes of abraded iron. The -560 mesh slime mineral was also saved.

By far the greatest proportion of mineral crushed to the -200 + 560 mesh range; in fact, it was difficult to obtain any appreciable quantity of sizes above 200 meah as merely the agitation of screening caused most of it to pass a 200 mesh sieve. Microscopic examination of a number of ores, and flotation plant products, containing jarosites , confirmed the tendency for the jarosite particles (when occurring in ores receiving the usual degree of grinding for practical flotation) to concentrate in this -200 + 560 mesh size range. Consequently, the major portion of flotation experiments were conducted on the -200 + 560 mesh sizes.

The ore obtained from La Plata, Utah, consisted principally of plumbojarosite. In the mine it occurred intimately mixed with

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[38] Over 40 individual settlings and decantations were necessary to remove the -560 mesh materials.

cerussite, anglesite, soft oxidized iron, and a very minute amount of incompletely altered galena. A fairly clean sample of about 100 pounds of plumbojarosite was obtained by hand sorting a leaser's stock pile. It was thought that a high grade plumbojarosite concentrate might be made for flotation feed by tabling. The galena, cerussite, and anglesite could be cut off high on the table and the plumbojarosite, with its low specific gravity, could be taken off from the tailing position. This procedure worked admirably for eliminating the free cerussite, anglesite, and galena but the jarosite product showed little change from the head sample (See Table II). Nearly the same high proportion of lead present as sulphide and carbonate was found as in the Mexican mineral. Also, as with the Mexican ore, the microscope revealed apparently nothing but very pure plumbojarosite crystals, the only difference being a lighter color and much smaller average crystal sizes. The tendency for this ore to break to slime size was so great that the agitation and washing on the Wifley table alone served to render it all -200 mesh size. Subsequent attempt to deslime the -560 mesh material demonstrated that the whole sample passed naturally into -560 mesh "slime" size. Thus no flotation feed, above slime size, could be secured from this ore. The lot was reserved for whatever future work might be done on slimes.

Although the Mexican mineral was the only one used to any considerable extent the above discussion of the minerals and their preparation for flotation feed has been taken up in detail because of

the emphasis it throws on the peculiarities of the jarosite composition and its ready tendency to crush to fine sizes.

TABLE II

Analyses of Minerals Used in Flotation Tests

Mineral and Source	% Pb as Oxide	% Pb as Sulphide	% Pb as Jarosite	% Pb Total	% Fe	% CaO	% S	$\%SiO_2$	% Ba	Oz. Ag	Oz. Au
Tintic Standard Mill Ore:											
Heads	2.70	0.33	1.20	4.14	5.92	1.38	2.2	28.0	0.0	17.4	Tr.
Sands			0.50	4.66						19.0	
Slimes			1.10	3.71						13.0	
Chihuahua, Mex. Plumbojarosite	1.22	5.30	9.84	16.4		0.36				2.7	
La Plata, Utah Plumbojarosite											
Heads to Table	3.32	6.86	7.91							1.4	
Conc. from Table	2.98	6.22	8.62								

EXPERIMENTAL PROCEDURE

Introduction

All flotation tests were made in a 50-gram University of Utah type mechanical agitation machine.[39] A celulloid walled cell and a bakelite coated propeller shaft were used with the exception of two special cases. Because of the limited supply of the pure plumbojarosite, all tests using this mineral were made with 25 instead of 50 gram charges. Preliminary tests demonstrated that the difference introduced by the higher pulp dilution when using a 25 gram charge, was very little and, since all tests were run with the same pulp dilution, correct comparisons could be made. Preagitation in each test was carried on at 1 to 1 pulp density. Time of preagitation was 4 minutes with exception of special cases. The collection period was governed by the time required to bring the total water used up to 300 cc. except where changes in frothing conditions prevented this addition. This resulted in a collection period of about 10 minutes duration. All reagents were added in solution with but two exceptions and all tests were run at room temperature with exception of some of the cases where fatty acids were used. Reagents soluble in water were added in water solutions of definite strength; those difficulty water soluble were added in alcohol solution. It has previously been stated that alcohol has no effect on the flotation

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[39]Gates and Jacobsen. Development and Operation of a 50-gm Flotation Machine. Eng. & Min. Jl., May 9, 1925.

other than possibly a slight increase in frothing.[40] However, the greatest work has shown that at higher concentrations the alcohol reacts with the celluloid cell and that camphor is one of the resulting products. Although this apparently has no effect other than the injury to the cell, it introduces a question and hence concentrations of reagents have been made such that factors of this kind were avoided as far as possible.

Frothing Agent

In keeping with the previous work conducted on the flotation fundamental program here at this station, terpineol was used as a frother throughout. This agent, an alcohol of the terpene group, has proven very satisfactory. It was originally selected because of its qualifications as a frother, its universal occurrence as an important constitutent of pine oils, its comparative cheapness, and its ready solubility in water.

Figure 1 shows the percentage of plumbojarosite floated by various concentrations of terpineol for different sizes of the mineral. One explanation for the recovery obtained on -200 mesh mineral is that it is purely mechanical. That is, there is a critical point in the surface-weight relation; as size of grain decreases, the amount of surface (plus the effect of the flaky micaceous character of the jarosite crystals) predominates over the effect of weight and as a consequence the grains are entangled and buoyed up in the plentiful froth

[40] Gaudin, A.M. Flotation Fundamentals, Part I. loc. cit. p-5.

developed by the terpineol. This explanation is backed by the facts that there are no reactions conceivable between jarosite and terpineol; a negative qualitative test was obtained of the tailing water in each case for iron, the most likely constituent of the jarosite itself, which would likely go into solution and enter into reactions.

The point in the size range where recovery begins is abrupt. To check this last point a -200 + 280 mesh product was prepared by sedimentation. Recovery was nearly as good on this size as it was on the -200 +560 mesh. The relation of recovery to size of grain is brought out more clearly by Figure 2, in which recovery is compared to size of grain when a constant amount of 0.5 pounds terpineol per ton is used. Table III gives the data used in Figures 1 and 2.

Another explanation for the recovery with terpineol is adsorption[41] of terpineol by the jarosite. Whether a reagent adsorbs by a

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[41] Adsorption: The term adsorption is one that has been widely (perhaps sometimes too widely) applied to explanations of flotation phenomena. Its use inthis respect has received criticism (see Taggart. Chemical Reactions in Flotation. Tech. Paper No 312, A.I.M.M.E. 1930). In this paper the term adsorption will be used in the same sense as adopted by Taggart in the paper just referred to,i.e.: as concentration at an interface between different phases in a heterogeneous system by action of some mechanism not thoroughly understood. Taggart goes further to say that: "the mechanism of the action of flotation reagents in solution leads toward the conclusion that so far as concentration of those reagents that directly determine collecting is concerned, the mechanism is invariably one of chemical reaction between the reagent and one of the constituents of the ore." However, in the present paper the meaning will not be narrowed to simple chemical action but will be considered as properly including such mechanisms as are suggested by the terms "residual or stray valance" and "electrostatic charge."

purely chemical affinity, a residual or stray valance, or an electrostatic charge attraction, and whether it is adsorbed on the mineral surface itself or in an enveloping gas film, are questions in the fundamentals of flotation that have as yet received no absolute proof or general acceptance. However, assuming that terpineol might adsorb on jarosite, then it is possible that a surface would present a sufficiently oily character to cause adherence to the air-water interfaces of the bubbles. This explanation is not invalidated by the abruptness with which recovery begins in respect to size of grain because one would not expect that the surface which adsorbed terpineol would produce would be especially efficient in collection property and hence the surface-weight relation would still come into active operation.

Enlarging on this, the combined effect of size-weight relationship and surface energy of mineral particle may be considered. It is well established that as particle size decreases, the surface energy increases. Conceding that terpineol may have a slight tendency for adsorption at the solid-liquid interface, then as particle size decreased there would be a point where the surface energy would

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4(continued) However, when in any particular case there seems to be sufficient proof to warrant it, an attempt will be made to enlarge on the general definition of adsorption and narrow the explanation as much as possible.

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become large enough to bring about adsorption. This adsorption may be only in the nature of a very incomplete monomolecular film depending on the intensity of the expression of surface energy at different points of the crystal according to its particular make-up. It follows, due to surface-weight relation, that the smaller the grain the less complete need be the filming to cause recovery; hence the increase in recovery of finer sizes but no recovery on coarser sizes when using terpineol alone.

A brief quotation from Fahrenwald[42] may aid in summarizing the factor of grain size in flotation:

"The factors controlling the size of a mineral particle that can be floated are: (1) The degrees of oil-mineral, oil-water, and air-water adsorption which determine the forcewith which the particle is held to the bubble; (2) the shape of the particle; (3) its specific gravity; (4) the cleaness of its surface, which influences the degrees of adsorption(1); and (5) the swirl of the pulp."

It may be well to add here that with certain minerals terpineol acts as an efficient collector. Work this year at this station by Messers Polisson and Lange on chalcopyrite and pyrite has demonstrated that nearly 100% recovery may be obtained on these minerals by use of terpineol alone.[43]

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[42] Fahrenwald,A.W. Idaho Bureau of Mines and Geology. Pamphlet No.2. Page 1. Moscow, Idaho, 1921.

[43] Polisson and Lange. Private Communication; work on Flotation of Chalcopyrite and Pyrite. To be published as a Technical Paper of the Dept. of Mining and Metallurgical Research, University of Utah.

A concentration of 0.5 pounds terpineol per ton of ore was chosen as a constant for future tests as the amount which gave the most desirable frothing conditions.

Flotation and pH Degree

Reference to Table III shows that in each case where terpineol and distilled water were used the pH was raised from pH 5.6 (the average value of the distill H_2O used) to a pH of 7.1. Thus, distilled water alone with the mineral furnishes a practically neutral circuit. Since there could be found no evidence of any solution of the jarosite itself it seems as though this must be attributed to the gypsum present. Qualitative tests for calcium ions and sulphate ions were both positive.

To determine the effect of an acid and an alkaline circuit on recovery, a series of tests were run using varying concentrations of common acids and bases. Figures 3 and 4 show the recoveries obtained (the upper curves show pH values of the circuit at corresponding concentrations). The tabulated data for these tests is given in Table IV. The effect on recovery as pH changes with the amount of reagent added is pronounced. A variation in the circuit on either side of neutral causes decrease which seems to conform somewhat to the magnitude of variation to pH. (It is not implied that pH value itself is a cause of flotation phenomena; rather it is merely a measure of the concentration of H or OH ions which may or may not be direct results of a

cause.

Plumbojarosite has been found to be slowly soluble in hydrochloric acid even in dilute concentrations. However, with the very weak concentrations of reagent and short time used this action was not sufficient to render a test of the tailing water positive for Pb-ion. A very faint positive test was obtained for iron when hydrochloric acid was used.

In attempting explanation for this depression there are several possible considerations to be made. If the recovery obtained by terpineol alone is purely mechanical, as suggested above, then whatever the change caused by the acid or base added its effect must be to change the character of the bubbles (that is, change the surface tension or surface charge relations) so that the bubbles are not capable of carrying the same weight of particles. As far as the eye could detect there was no appreciable change in the froth except on higher concentrations of the acids, when the froth was materially decreased.

If, on the other hand, recovery is considered as caused by an adsorption of terpineol, then any chemical action of reagents on the surface of the mineral might alter it so that terpineol would not adsorb or since these reagents are all electrolytes, they might alter the attraction relations (conceding electrical effects in the adsorption of terpineol).

Aside from these explanations is the possibility that salts

might be formed by the interaction of the reagents and the mineral which would act as depressants. For instance, in use of hydrochloric acid, ferric chloride may be produced and this has been found to depress in many cases.[44] Also, it is to be remembered that a small amount of gypsum is present and hence calcium salts[45] would probably be formed in most cases.

Effects of Other Inorganic Salts

It has been stated previously that in this work more attention has been paid to the study of collecting than to modifying agents, except in the case of sulphidizing agents, if these be considered modifying. Besides the acids and bases used, which have been discussed in the preceding section, a few inorganic salts have been used in particular combinations in attempts at certain desired results with the other reagents studied. A separate section will not be devoted to these modifying agents but they will be discussed under the heading of the particular type of reagent with which they were used.

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[44] Gaudin, A.M. Flotation Fundamentals, Part I. loc. cit. p-44
[45] Gaudin, A.M. Flotation Fundamentals, Part 2, loc. cit. p-24
Idem, Part 1, p. 42

Sulphidizing Experiments
Employing Reagents in Solution

Introduction: In the introduction to this paper, stress has been laid on the importance of sulphidizing in the flotation of oxidized ores. Practically all plants operating on oxidized metallic ores and many plants in which some of the ore is partially oxidized employ sulphidizing agents. By far the most widely used sulphidizing agent is sodium sulphide.[46] Reference has been given on page 11 to the poor results indicated in attempting to sulphidize.

Considerable experimentation has been done to determine the relative efficiencies of various sulphidizing agents.[47] The more powerful of these were selected for the present work. The first series of tests were made using aqueous solutions of these agents. Those employed were: hydrogen sulphide, ammonium sulphide, and sulphureted pine oil.[48] Table V gives the data obtained with these alone and in combination with other reagents.

Theoretical Discussion: Although concentrations of sodium sulphide as high as 30 pounds per ton were used and preagitation periods up to 60 minutes employed, there w s no visible sulphidizing effected. However, it is evident that some change is brought about by the presence of the sulphidizing agent as reflected by its strong depressing actions, even

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[46] Sodium sulphide used on oxidized lead ores only in 1928 amounted to 159,666 lbs. Total sodium sulphide used in 1928 amounted to 2,399,239 lbs. Miller and Kidd. Flotation Reagents,1928.U.S.Bur.Mines,R.I.Serial 3004.

[47] Leaver and Lawrence. Barium Polysulphide in Sulphidizing Oxidation for Flotation. Report of Investigations,Serial 2698,U.S.Bur.of Mines. 1925.

[48] Sulphureted Pine oil has been considered to have a sulphidizing action--(See Lyon and Ralston. Innovations in the Metallurgy of Lead. U.S.Bur. of Mines, Bull. 157, 1918, p-105).

when used in small concentrations and for short periods of contact. Using a constant amount of sodium sulphide, the extent of depression increased with increase of preagitation time (See Figure 7). Since there is no visible sulphidizing effected and no other chemical changes manifest, it would seem that fundamental explanations of this depressing action can not be, at present, narrowed beyond those brought up in discussing the action of terpineol.

A.M.Gaudin[49] states that it has been determined that the difficulty in sulphidizing certain oxidized lead minerals is largely a mechanical one, certain coatings being adherent while others are loose. In offering a chemical explanation for this, he takes into consideration the equilibrium between the solubility product of the salt constituting the mineral, the solubility product of the sulphide formed and the solubility product of the salt resulting in solution from the exchange adsorption. In this light, the ease of sulphidizing lead carbonate as compared with lead sulphate is explained by the fact that the $CO_3^{=}$ ions are easily removed by the H^{+} ions but the concentration of $SO_4^{=}$ ions will quickly up to prevent the further replacement of $SO_4^{=}$ ions by $S^{=}$ ions at the surface of the mineral.

Because of this it is imperative, Gaudin states, if lead sulphate is to be made easily sulphidizable, to eliminate sulphate ions as soon as formed. Or, another method would be to coat the lead sulphate with a lead salt that is readily sulphidized, such as lead carbonate. To

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[49] Gaudin,A.M. Flotation Mechanism, a Discussion of the Functions of Flotation Reagents. Tech. Pub. No.4, A.I.M.M.E., 1927, p-22.

this end he suggests the use of ammonium carbonate, to be followed by immediate sulphidizing.

This view deserves consideration in the case of plumbojarosite since the mineral is a sulphate. It was with this in mind that the tests (Table V) were made using ammonium carbonate prior to sulphidizing treatments. If the reactions above presented develop in the case of plumbojarosite, then such an explanation may be offered for the difficulty met with in attempting sulphidizing of the mineral. To add to the retarding effect of the sulphate ions from the plumbojarosite, is the influence of additional sulphate ions due to the small percentage of gypsum present. This concentration of $SO_4^=$ ions may even require, as pointed out by Gaudin, a higher concentration of sulphide ions than is possible from the dissociation of hydrogen sulphide in acid or neutral circuit. (it will be noted in Table V that the circuit was alkaline in each case).

However, it was found that marked depression resulted in every test employing the suggested combination with plumbojarosite. In considering this it is well to remember plumbojarosite is a basic sulphate and hence highly insoluble. While this may mean that the plumbojarosite gives up no $SO_4^=$ ions, it also suggests that the Pb atom may be protected from the desired action with the ammonium carbonate. Again, were it available, it is but one atom against 50 atoms of iron, oxygen, hydrogen and sulphur in the molecule (see formula page 16). Hence the reason for depression probably goes back to the

same unfinished explanation given for the result with the sodium sulphide above.

Effects of Other Reagents: The possibility of a sulphidizing or filming which is not visible to the eye plus the fact that the xanthates have come into wide use as collectors[50], led to the tests (Table V) employing xanthate subsequent to sulphidizing treatment.

Copper sulphate and ferric chloride were used in combination with the sulphidizing agents in the hope that they might promote a sulphide filming of the mineral. Ferric chloride brine solution (of the same strength as employed in the method for plumbojarosite analysis for extraction of sulphide lead) was used preliminary to the sulphidizing agent in the hope of exposing lead to the sulphidizing action. The poor results obtained might have been due to the excessive quantities of salts present in the brine solution.

Speaking only of the tests for which sulphidizing reagents were added in solution: sodium sulphide showed no visible sulphidizing effect after one hour treatment, ammonium polysulphide showed a slight darkening effect after one hour, and hydrogen sulphide showed no great degree of darkening up to 3 hours, but was blackened after 4 hours of contact. In every case of each of the above for different concentrations, different periods of preagitation, and different combinations, there was depression as compared with the recovery obtained with terpineol alone.

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[50] The use of the xanthatees, amyl in particular, has been advantageously extended to the field of oxide flotation in connection with sulphidizing.

Sulphureted Pine Oil: Sulphureted pine oil has been considered here along with the sulphidizing agent because it has, in the past, been described as a sulphidizing agent.[51] However, since it proved to be a very efficient collector for plumbojarosite when used only with terpineol and thus very different in its action than any of the other sulphidizing agents used, it seems very probable that is has a different function. It showed a marked reaction with the celluloid cell walls, leaving a yellow stain which resisted almost all attempts of removal and which increased recovery on subsequent tests until completely removed. Since it is an active complex organic compound, there was a possibility of interaction with the celluloid cell that might have produced a secondary product which in turn caused the high recovery of plumbojarosite. To check this possibility, test number 95 was made using an iron cell coated with aluminum paint. A 100% recovery was likewise obtained in this cell; conditions of froth and time of collection were similar to the previous case. However, its ready-collecting properties are easily destroyed by additions of acid or base. (See Figure 8).

Interesting in considering an explanation for this is the statement of Bains:[52]

> "The remarkable phenomena produced by extremely small amounts of coal-tar derivatives and other reagents not classified as oils are more dependent on the electrostatic than on the interfacial tensions unless one is willing to consider these interfacial tensions as electrostatic, also."

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[51] Lyon and Ralston. Loc. cit. p.105

[52] Bains, T.A. Electrometer Tests in Flotation. Eng. and Min. Jl., vol. 117, 1924, p-885

Sulphidizing Experiments with Dry Hydrogen Sulphide Gas

The results of attempted sulphidizing employing dry hydrogen sulphide gas, instead of sulphidizing agents in solution, are still more interesting. Hydrogen sulphide used as the gas on dry ore has been given a good deal of consideration in the early attempts at sulphidizingoxidized ores,[53] and has been the subject of many patents. It has been found powerful in its action and since it can be cheaply made,[54] it can have commercial application. The rate of diffusion of a gas in the dry state is very much greater than that of the same gas in solution. Also, it is a well known fact that gases adsorb more or less tenaciously on minerals.[55] This last characteristic is one which promises to be of considerable interest in the fundamentals of flotation phenomena.[56] It was because of these facts that a few tests were made (See Table VI) using dry hydrogen sulphide gas for sulphidizing.

A simple apparatus for treating a charge of ore prior to flotation was made and employed in the following manner. A tray, carrying a filter paper, was fitted snugly into a Buckner funnel set in a suction flask. The mineral was spread on the tray; a rubber cloth cap clamped tightly over the top and hydrogen sulphide gas (dried by passage through sulphuric

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[53] Lyon and Ralston. Loc. cit. pp.88-93

[54] Ibid.

[55] Freundlich,H. Colloids and Capillary Chemistry. Dutton & Co.N.Y. 1922 p-102-155

[56] Adams,A.S. Gas Adsorption in Flotation. Tech.Pub. No.41,A.I.M.M.E.,1927

acid), passed in through a valve in the rubber cap. The gas was drawn down through the ore bed by suction.

Thirty minutes of such a treatment produced only a barely visible change; 90 minutes, a notable darkening; 2 hours a blackening; and 4 hours changed all particles to a glistening black. That the action of hydrogen sulphide as the gas is more decidedly pronouncee than when used in aqueous solution is evident since it required over three hours contact with a saturated solution to produce any appreciable darkening.

Despite the black appearacne of the mineral (after the hydrogen sulphide gas treatment) when viewed in mass by eye, examination under the microscope showed that no true sulphidizing action had taken place. The crystals appeared in their original color and luster except for a slight smoky cast when in aggregation. The black appearance was due to accumulation of black material, probably principally excess sulphur, at the boundaries of crystals occurring in the characteristic platy aggregates. Had the mineral been truly sulphidized, recovery should have been increased in some of the subsequent flotation tests (see Table VII). However, in every case, (except that in which oleic acid was used as a collector) depression resulted. Also, as in the case of sodium sulphide (see Figure 7) increase in time of sulphidizing treatment caused a correspondingly greater degree of depression in the subsequent flotation data.

Effects of Other Reagents: Thiocarbanilid, a derivative of coal tar containing sulphur and having a powerful collecting action similar to the xanthates, was used subsequent to the sulphidizing treatment. This reagent is considered one of the very best collectors known.[57] However, on

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[57] Weinig and Palmer. The Trend of Flotation. Colorado School of Mines Quarterly., vol. XXIV, 1929.

plumbojarosite which had been given the hydrogen sulphide treatment the collecting action of thiocarbanilid was nil.

It is interesting to note that practically no recovery is made with sulphereted pine oil subsequent to the hydrogen sulphide treatment and yet the recovery obtained by oleic acid is the same as obtained on the untreated ore (see Tables VI and VIII). The case of oleic acid is the one exception. Although each of the other collectors tried, subsequent to sulphidizing, were strong collecting reagents, they did not have any beneficial effect over the almost complete depression caused by the hydrogen sulphide treatment. That oleic acid should act so differently in this case indicates that the adsorbed gas film plays an important role.

There is much reliable evidence that many gases are adsorbed at the solid gas interface in layers of several molecules thickness.[58] If there is such a complete and tenaceously adsorbed gas film on the mineral surface, that expression of the surface energy forces of the mineral itself are satisfied in binding the film and thus have no outside field of influence , then it is unlikely that oleic acid molecules should adsorb and sulphureted pine oil molecules not (whereas both are highly efficient on the untreated ore) unless there is a very different reason for the flotation with these reagents . With oleic acid the mineral bearing froth is in the nature of a thick coagulation (this will be discussed in the following section). This indicates that there are capillary forces at play causing flocculation. Evidently these capillary forces are in no way changed by the hydrogen sulphide as in the case of oleic acid. On the other hand apparently such forces do not act for

flocculation with sulphureted pine oil and the action, whatever it may be, that it ordinarily has for collection of the mineral, is inhibited[59] by the hydrogen sulphide.

Theoretical Considerations: The part that adsorbed gases may play in the fundamentals of flotation, is a subject which has been recognized as important,[60] but it presents a difficult field of investigation and there has been as yet no certain fundamental explanations developed. In the paper above referred to, Adams concludes from experiments on a variety of minerals that flotability depends on the nature of the gas in the bubble and that the internal pressure of the gas at least partly accounts for the attachment of the mineral to the bubble. When the mineral particle is subjected to a desorption treatment, a marked change in flotability results. This is accounted for by considering the extreme variation in surface energy at different points on the mineral particles, due to edge and point effects.

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[58] Freundlich. loc. cit. p-122

[59] Freundlich. Colloid and Capillary Chemistry. Dutton & Co. New York 1922. pp. 146-154

[60] Adams, A.S. loc. cit.

Because of this, neither the desorption or change in flotability is constant.

Again, aside from explanations of the phenomena related directly to adsorbed gas is the possible influence of colloidal material--sulphur.[61] Hydrogen sulphide is a strong oxidizing agent. Ferrous sulphate reduces hydrogen sulphide. It is a question whether the structure of jarosite is such that ferrous sulphate could be available for this reaction. If it were, then there is possible a development of colloidal sulphur. Lyon and Ralston have found that colloidal Sulphur does not sulphidize lead carbonate and that poor results are obtained in its presence.

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[61] Lyon and Ralston. loc. cit. p. 105

Flotation with Sodium Oleate and the Fatty Acids

The use of the fatty acids in flotation: Previous research[62] has demonstrated that oxidized minerals can be effectively floated with fatty acids of both the saturated and unsaturated series.[63] Also silicates[64] and sulphides[65] have proven floatable with this type of reagent. The use of fatty acids as collecting agents is now being introduced in practice,[66] and so some of the fatty acids, (selected to represent a wide range in number of carbon atoms, in both the saturated and unsaturated series) were studied as potential collectors for plumbojarosite.

Flotation with Sodium Oleate: In past work oleic acid has proven especially efficient and therefore sodium oleate has also been given consideration. The explanation for the collecting action of fatty acids has largely been made on the basis of the formation of a metal soap.

Comparison of recoveries on various sizes of plumbojarosite with sodium oleate and oleic acid is shown in Figure 9. Their efficiencies are similar. Sodium oleate is slightly more efficient than oleic acid on minus 200 plus 560 mesh mineral but the reverse is true on the coarser sizes. Sodium oleate hydrolyzes in water to sodium hydroxide and oleic acid so that its effect should be similar.[67]

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[62] Gaudin and Hansen. loc. cit. p-57.
[63] Gaudin and Martin. Flotation Fundamentals, Part 3. loc. cit. p-7.
[64] Glover. loc. cit. p-80
Iverson. loc. cit.
[65] Gaudin. Flotation Fundamentals, Part 1, loc. cit. p-8
[66] Weinig and Palmer. loc. cit. p-24

The effect of inorganic acids and bases was a reduction in recovery. This is shown in Table VIII.

Lead nitrate was also tried with sodium oleate because in the work above referred to, Mr. Iverson obtained remarkable recoveries on silicate minerals with this combination in low concentrations of reagents. This he ascribes to the formation of the lead soap. In the case of plumbojarosite nothing was proved by this test because 100 per cent. recovery is obtained with a very low concentration of sodium oleate and the presence of lead nitrate does not change this.

Flotation with Oleic Acid: Since there was little difference in the results obtained with sodium oleate and oleic acid, and because it was desirous to make comparisons with other fatty acids, oleic acid was used principally in subsequent tests. Table IX gives data on the flotation, with oleic acid, of various sizes of plumbojarosite, including "Slimes". Figure 10 shows the effect of grain size on recovery when using 0.05 pound oleic acid per ton with 0.5 pound terpineol per ton as frother. This graph (Figure 10) brings out strikingly the effect of the vastly greater surface developed in the slime sizes.

On minus 200 plus 560 mesh, plumbojarosite , a 100% recovery is made with only 0.05 pound oleic acid per ton. For this concentration an adsorbed monomolecular film would probably be less than complete. Gaudin[68]

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[66] DeVaney and Clemmer. Floating Carbonate and Oxide Manganese Ores. Eng. and Min. Jl. , Sept. 28, 1929, p-508

[67] Gaudin and Martin. Flotation Fundamentals, Part 3, loc. cit. p-10

[68] Gaudin Flotation Fundamentals, Part 1, loc. cit. p-20

has calculated (in the case of galena), for about the same size range as used above, that a concentration of oleic acid of about 0.06 pound per ton would be necessary for a complete monomolecular film. Because of lack of data on jarosite any such calculation would be very approximate for this mineral. However, considering the thinmicaceous character of its crystals, it is certain that the surface for jarosite would be much larger than that for galena and a correspondingly greater concentration of fatty acid would be necessary. In the coarser sizes, the increased ratio of weight to surface of the mineral particles overcomes the ability of the incomplete film to float the grains,while in the slime sizes the vastly greater surface area overbalances the ability of a less complete film to cause recovery.

High concentrations of the fatty acids caused a slight lowering in recovery due to a change in the character of the froth. At a concentration of 2.0 pounds of oleic acid per ton a viscous "skin", heavily laden with mineral but poor in froth bubbles, formed at the surface of the pulp. This may be due to action with the terpineol; an effect of the higher concentration of alcohol; or merely an increased thickness of film of oleic acid at the water surface due to the excess of the same.

Theoretical Consideration: A great deal of evidence has been obtained that flotation with the fatty acids depends upon an adsorption of the molecules oriented with the polar ends drawn against the mineral surfaces and the non-polar ends presented to the surrounding pulp solutions. Thus the mineral obtains a non-polar coating. The polar end of the molecule is represented by the more active and more soluble carboxyl group, while the non-polar end comprises the more insoluble hydrocarbon chain. The non-polar coating formed by this orientation is not wetted by water and

is drawn into the gas-liquid interface of the froth bubbles and buoyed to the surface.

There seems to be a strong tendency towards flocculation with the fatty acids. The whole pulp contains flocculent aggregates, the flocs appearing to be formed by adhesion of the greasy filmed particles to each other as well as to the bubbles. Flocculation is considered detrimental to good flotation.[69] (A brief discussion of flocculation phenomena is given on page 65).

The reason for adsorption of the fatty acid molecule on the mineral surface is largely ascribed to the formation of an insoluble metal soap. The higher the fatty acid in the series the more insoluble the resulting soap. Hence, less of the reagent is needed because the coating is more permanent. Adsorption at the surface of the mineral particles is considered as being in dynamic equilibrium with the amount of reagent in solution. That is, flotation with the fatty acids essentially depends on the solubility relations of the metallic soaps which are adsorbed at the surface.

In the case of pure plumbojarosite, the only metal ions available would be those of iron and lead. With the mineral used, calcium would also be present due to the small amount of gypsum contained. The attachment of the fatty acid molecule may perhaps be made merely by the influence of the residual valence at the surface of the mineral and thus no ion necessarily displaced. Also, if the attraction and orientation of the collector molecule is due to some strictly capillary force or electrostatic charge, then again there would not necessarily be a free ion in

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[69] Smith and Pickett. Flocculation in Flotation. Eng. and Min. Jl., Vol.107 pp. 365-6 (1919)
Hahn, A.W. Flotation of Oxidized Lead-Silver Ores. loc. cit. p.2

solution, which a qualitative test would confirm.

At any rate, the number of variables entering in explanation of the phenomena could be limited if a positive or a negative test could be made for any ions which might be expected to be in solution due to the mechanism of the process. It has been mentioned that negative tests were obtained for the presence of iron and lead in solution when the pulp is agitated in water alone or with terpineol added. This also holds true after agitation with the fatty acids. The presence of sulphate ion obviously would mean the most in explaining the adsorption of the fatty acid on a chemical basis. Although the gypsum is fairly insoluble it is much more apt to be soluble than the constituents of the jarosite. However, the presence of gypsum interferes with testing for sulphate ion which would be known to come from the plumbojarosite itself. To get at this it would be necessary to eliminate the gypsum.

Elimination of the Effect of Calcium: Also, with the gypsum present it is possible that the formation of calcium oleate would take place and this might be the coating on the mineral, causing recovery. Although calcium oleate is more soluble than either lead or iron oleate, there is no proof that it is not a cause for the flotation unless all calcium were previously removed. In discussing preparation of the mineral mention was made of the impossibility of completely eliminating gypsum by means of any gravity concentration. It would be necessary to completely eliminate the gypsum to prove the above point because so very little fatty acid is required for complete recovery and hence a very little calcium is necessary. Any chemical means for removing the gypsum is questionable because it

might effect of the surface of the jarosite which in turn might cause a considerable difference in flotation results and hence afford no basis for comparison. Several leaching tests were made in which it was hoped the gypsum could be eliminated without changing the jarosite mineral surface. Both the residue and the leach solution were analyzed in each case for calcium and also for the constituents of the jarosite.

Several different leaching reagents and combinations were tried. With but one exception these methods depended upon subsequent solution of calcium carbonate (to which the gypsum had been converted) with dilute hydrochloric acid. It had been previously thought that cold dilute hydrochloric acid had no effect on plumbojarosite. However, it was found that a leach with hydrochloric acid sufficient to remove all calcium carbonate also took a slight amount of iron and lead into solution. Undoubtedly there was at least a surface change. Subsequent flotation tests gave very erratic results. Although these developments were interesting they are aside from the problem and therefore none of this data has been included in this paper. The one leaching method which seemed reliable was that employing ethyl alcohol (See Table X). The action seems to be a simple solvent action of the alcohol on the calcium sulphate. Although for final removal of gypsum the leach was carried out for 54 hours, there was no solution of the jarosite constituents as proven by the most sensitive qualitative tests; nor was any change visible by microscopic examination. Subsequent flotation of mineral treated in this manner showed no material change when using oleic acid, from results for corresponding tests on the untreated mineral. Thus it was proved that the calcium

present was not the cause for flotation with the fatty acids.

Since sulphate ions due to the gypsum were now removed tests could also be made to check the presence or absence of sulphate ions from the jarosite which might possibly be displaced by adsorption of the fatty acid. The qualitative barium chloride test was used and gave negative results.

Flotation of Slime Size Plumbojarosite with Oleic Acid

Since oleic acid has proven to be a powerful collector for plumbojarosite it was employed as collecting agent for tests on slime (minus 560 mesh) plumbojarosite. The fact that in many jarosite bearing ores the jarosite mineral naturally occurs in slime sizes, and in any case readily crushes to slime sizes, must not be overlooked. The effect of slimes in flotation is very important. It is not only difficult to obtain an efficient differential separation from slimes themselves, but the presence of slimes is detrimental to good flotation in general.[70] That makes this phase of the problem one of considerable importance.

The data obtained on slime flotation is presented in Table IX. One of the major effects of slimes is the large consumption of reagents due to the extremely great surface area exposed. This is brought out by Test No. 150 which shows that for complete recovery of slimes 0.5 pound oleic acid per ton is necessary as compared with only 0.05 pound per ton on minus 200 plus 560 mesh mineral. It is also interesting to note that

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[70] Weinig and Palmer. Loc. cit. p-41

concentrations of 1.0 pound per ton of sulphuric acid and sodium carbonate, respectively, when using 0.05 pound of oleic acid per ton, cause very slight depressions; whereas, on minus 200 plus 560 mesh mineral, they caused almost complete depression. Comparison of recoveries on slime size La Plata ore shows almost the same values for concentrations of oleic acid. Some explanation of the above results and the phenomena that occur in connection with slimes in flotation may be had by going into a theoretical discussion. However, this phase of the problem belongs more strictly to the separation experimentation in connection with typical oxidized ores, which has been started and is being planned as a separate work not to be included in this paper. It will suffice to say at this point that more complex conditions are introduced in the realm of slimes--more intense surface energies come into play; electrical relations become of great importance; and colloidal phenomena and capillary forces introduce intricate fundamental considerations. The papers of Moses, Hahn, and Fahrenwald,[71] present many of these problems in their practical, and theoretical relations. Also the classical works on colloidal chemistry, a few of which are listed on page 48, presents the foundations of the phenomena involved.

Comparison of Flotation with Different Fatty Acids at Room Temperature: A list of the fatty acids of both the saturated and unsaturated series which were used in this work is given in Figure 11. Three of these acids, crotonic, lauric, and stearic, have melting points well above room temperature.

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[71] Moses, F.G. loc. cit.
Hahn, A.W. loc. cit.
Fahrenwald, A.W. Surface Reactions in Flotation. Trans.A.I.M.M.E. No. 1283-M, Jan. 1924.

Melting points of the fatty acids used are as follows:

Saturated Series		Unsaturated Series	
Name	Melting Point	Name	Melting Point
Heptylic	-10.0°C	Crotonic	72.0°C(α)
Lauric	48.0	Undecylenic	24.5
Stearic	69.3	Oleic	14.0

There is much experimental evidence that temperature[72] has a pronounced effect on recovery when using fatty acids which are solids at ordinary room temperature. With such acids there is a marked increase in recovery for a certain amount of reagent with increase of temperature and the effedt is greatest near the melting point of the reagent. This is most probably due to the greatly increased mobility with rise of temperature and hence greater dispersion prior to adsorption. Also because of the insolubility of the acids in water, they were dissolved in alcohol,but on addition to the pulp the alcohol solution is greatly diluted with water and probably some of the acids, especially the higher ones, would precipitate out. Increased temperature would tend to correct this.

To properly compare results,with different fatty acids, the tests should be conducted with the pulp at the melting points of the acids. However, a series of tests, (Table XI and Figure 11) was first made for each acid at room temperature. This was done not only to furnish data for comparison with results at higher temperatures, but also, if any commercial application of these reagents in this type of ore were to develop,

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[72] Gaudin Flotation Fundamentals, Part 1, loc. cit. p-8
DeVaney and Clemmer. loc. cit.

it is more probable (especially in the localities in which this kind of ore would be treated) that the flotation circuit would be maintained at ordinary temperatures. Figure 11 is a direct plot of the results obtained with the six acids used. The curves show a general conformity to the rule that with homologous series of compounds, as the length of the hydrocarbon chain is increased, the amount of reagent to give a certain recovery is decreased. However, the usual regularity of difference as the series is ascended is lacking and in the case of lauric acid and stearic adids the positions are reversed. This discrepancy may be answered by the fact that stearic acid has a melting point of 69.3 degrees centigrade, whereas the melting point of lauric acid is 48 degrees centigrade,[73] although the difference seems strikingly large.

Another reason for the irregularity is that concentrations expressed in pounds per ton do not represent differences in numbers of molecules and since the gaps in the homologous series between the acids used are rather wide, this factor is noticeable. Consequently, the concentrations used were converted to millimoles of reagent per ton and so plotted. Figure 12 represents a direct plot by this method and shows the more uniform spacing of the curves. Figure 13 represents the same data in a logarithmic plot which makes the relations still more pronounced. The discrepancies due to melting point differences are, of course, not corrected by these plots.

Comparison of Flotation with Different Fatty Acids at Melting Point Temperature: Interesting difficulties arose in running the tests on the

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[73] The melting points and molecular weights of these acids were taken from the; International Critical Tables. McGraw Hill. New York.

three acids (crotonic, lauric and stearic) at their melting point (see Table XII and Figure 13) Complete recoveries were obtained with surprisingly small concentrations--for instance, lauric acid at a concentration of 0.005 pound per ton; for stearic acid, at room temperature it was necessary to use a concentration of 0.5 pounds per ton to obtain a recovery of 100%, but at melting point temperature a concentration of .005 pound gave complete recovery. These great differences (in order of the one one-hundredth the previous necessary concentrations) coupled with the fact that such small concentrations would represent very incomplete monomolecular films, led to a consideration of other variable factors which might be exerting an influence at these high temperatures.

The question was presented of the effect of high temperatures of the cell walls, the bakelite on the propeller shaft, and the terpineol. Test No. 194 shows that the bakelite did account for at least part of the recovery increase. With no frothing agent it could not be expected that a high recovery would be obtained; yet the few bubbles that did form were immediately coated with mineral so that a 22 per cent recovery was obtained in spite of the absence of a collector.

The first tests were run in the standard celluloid walled cell used throughout the previous work. The cell and the pulp were gradually heated to the temperatures of the respective melting points of the acids used and the test run with the cell resting in a water bath at a temperature slightly above that desired. Up to temperatures of 50 degrees Centigrade, no effect on the cell was observable. Above 70 degrees Centigrade the cell became pliable, although no other ill effect was noticed. It was thought unsafe to carry temperatures higher.

After each temperature run it was noted that the bakelite coating on the propeller shaft had undergone a change, had lost luster and become brittle. This change as noted above contributed a 22 per cent rise in recovery.

Test No. 192 proved that the terpineol contributed practically nothing, to the increase in recovery.

To check the possible effect of the celluloid cell walls, tests No. 188 and 193 were made using an iron cell coated with aluminum paint.

To check the effect of the metal cell tests No. 193 was run using only terpineol and a temperature of 65 degrees centigrade--this gave 100 per cent recovery in one minute. Tailing water of both of these runs gave negative tests for iron in solution. Thus the uncoated iron propellor apparently contributed nothing to the recovery. Only one difference is left--that of the metal cell wall as compared with the celluloid cell walls--to account for the great increase of recovery.

Since this cell was thoroughly coated with aluminum paint; since the presence, in the pulp, of soluble iron could not be positvely proved; and, since there were present no other reagents than terpineol with which to obtain a chemical reaction (and terpineol has been shown to have little or no effect), there seems to be in this case, little evidence on which to attribute the recovery to chemical reaction. No definite explanation is advanced as an alternative. However, during these tests the following thought has suggested itself which may be of no significane and yet it may be considered worthy of future investigation. Bakelite and celluloid are both non-conductors. When the propeller shaft is uncoated there is direct contact from the pulp through the shaft and bearing to the frame of the machine, while the celluloid is insulated from the frame. On

the other hand, when the shaft is bakelite coated the pulp is completely insulated from outside contact. When the metal cell is used there is contact with the frame though both the cell and the shaft--in other words, a closed circuit relation. These relations seem of importance if, as indicated, electrical phenomena play a part in the recovery peculiarities discussed above.

When the above factors which influenced recovery at high temperature are considered, it is obvious that the small concentrations of fatty acids used, played only a minor part in the recovery. Considering also, the trend of change in the curves of the fatty acid recovery under question with rise in temperature, it seems probable that could all variables other than purely the effect of the fatty acid under consideration be eliminated, agreement with the general rule for flotation with homologous series of reagents would result. However, any attempt to establish proof under these conditions would be questionnable because of the large and indefinite effect of these variables. Hence,no further tests were run. The three single tests for the three single acids in question were plotted in Figure 13, because they do give at least an idea of the great change caused by increase of temperature.

Flotation of Synthetic Mixtures of Plumbojarosite and Calcite Using Oleic Acid

Introduction: From the practical standpoint one of the major considerations in a flotation problem is the efficient separation of the valuable mineral from the gangue minerals with which it is associated. Plumbojarosite occurs with both siliceous gangues (for instance, the Tintic Standard ore) and calcareous gangues (for instance the La Plata ore).

A comprehensive investigation of the separation of the jarosite minerals from such gangues would probably assume the proportions of an independent research problem. The practical importance of this problem, however, demands at least a preliminary experimentation along this line in the present problem. With this in mind, a few separation tests have been made on synthetic mixtures of calcite and plumbojarosite and quartz and plumbojarosite.

Mixtures of Sized Minerals: Flotation of calcite has been described.[74] Some of the pure mineral that was used in this work by Hansen was obtained. Because the fatty acids gave high recoveries on the jarosite mineral, but at the same time are also collectors for calcite, it was thought wise to investigate the possibilities of separation of these two with a fatty acid (oleic acid was used). The charges were made up of 12.5 grams plumbojarosite and 12.5 grams of calcite.

In Hansen's work he has used, where possible, no frothing agent,

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[74] Gaudin and Hansen. Flotation Fundamentals

depending upon the froth forming power of the acid. To obtain comparable results a few preliminary tests were made on calcite alone (Table VIII and Figure 14).

A minus 100 plus 150 mesh product was used in combination with minus 200 plus 560 mesh plumbojarosite. This size ratio was selected for two reasons--first it was thought, although calcite is a soft mineral, that the natural tendency of jarosite to crush to the finer sizes would cause some such ratio in sizes of the two minerals; secondly, it was thought that comparisons could be made by using synthetic mixtures in which there was a gap in screen sizes between the minerals; screening the products; reversing the sizes used; screening the products and finally averaging the weights obtained for each. These results are shown in Table XIV.

This method did not prove very satisfactory since the range in sizes covers that in which jarosites begins to float whereas calcite floats in nearly a straight line relation through this range. Also, when plumbojarosite was used in the coarser size there seemed to be a tendency for it to be broken to some extent during the process, so that a portion of the reuslting concnetrate passed through the 150 mesh screen. Hence only a few tests were run in these combinations.

Figure 14 shows that the minus 200 plus 560 mesh plumbojarosite floats much more readily than the minus 100 plus 150 mesh calcite, there being a maximum difference of about 75 per cent at a reagent concentration of 0.05 pounds per ton. However, when combined, the calcite floated almost equally well. This is due to flocculation

effects. When plumbojarosite is floated with a fatty acid, large flocs start forming in the body of the pulp and rise to the surface in masses. When calcite (which shows the same tendency) is present, it is mechanically entrained by these flocs.

Flocculation Phenomena: It seems most probable that the flocculation phenomena has the greatest effect in the reduced recovery of plumbojarosite and increased recovery of calcite, but there should be also considered, the possibilities (1) of coating of the jarosite particles with calcium carbonate (jarosite has a slightly greater hardness than calcite); (2) of the depressing action of calcium salts such as lime (CaO).[75] (See Figure 4).

Flocculation is an important phenomena that is much discussed under the head of colloid or capillary chemistry.[76] Flocculation, or coagulation, may be briefly and generally defined as: The aggregation of the particles of the disperse phase to secondary units of different size and structure.[77] These units may be either reversible or irreversible. The terms flocculation and coagulation are generally interchangeably used although flocculation is perhaps more often applied to larger aggregations such as in the case under discussion. The electrokinetic forces come into important play in coagulation phenomena. Hence, addition of electrolytes or any change which alters the so-called

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[75] A discussion of the effect of CaO is given by Gates and Jacobsen. Flotatinn Fundamentals and their Practical Application. loc. cit.

[76] Freundlich. loc. cit. p-415.
Rideal. Surface Chemistry. Cambridge University Press, 1923. p-273
Svedberg, The. Colloid Chemistry. Chem. Cat. Co., N.Y. 1924.
Colloidal Symposium Monograph. (5 vols.) Chem. Cat. Co. N.Y. 1923-28
Moses, F.G. Colloids and Flotation. Tech. Paper, U.S. Bur. of Mines '18

[77] Svedberg. loc. cit. p-209

[78] Freundlich. Elements of Colloidal Chemistry. p-148

affect the arrangement of this electridal surface layer, or may discharge it to a certain degree, or may even oppositely charge it, and hence render conditions such that the particles are attracted or repelled, or else have no force working against their coalesence oncethey come into contact.

Peptization is the process of reversing coagulation. The mechanism of peptization is discussed clearly by Freundlich.[78]

The above paragraph on flocculation applies especially to the realm of colloids. However, these effects come into any consideration of surface phenomena and should be considered in amy attempt at explanations of the fundamentals involved. Especially is this true as smaller and smaller particles'size is considered.

Sodium silicate is a good peptizing or deflocculating agent and is used rather widely for this purpose, especially in oxidized ore flotation.[79] It keeps the gangue particles in a dispersed condition, so that they do not interfere with the action of the other reagents and do not collect in the bubble films.[80] One of the most probable explanations for the action of sodium silicate is on the basis of its colloidal nature, that it acts as a protective colloid. It is a lyophilic sol and as such probably adsorbs on the gangue particles and presents a surface to which water is attracted.

Hence sodium silicate was used in this work in an effort to obtain better separation between plumbojarosite and gangue minerals. Table VIII shows the effect of sodium silicate on calcite alone and the same is given

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[79] Hahn,A.W. Flotation of Oxidized Lead-Silver Ores. loc. cit.
[80] Weinig and Palmer. loc. cit. page 24.

for plumbojarosite in Table V. Figure 15 shows the effect on synthetic mixtures using coarse calcite and fine plumbojarosite. Maximum separation under these conditions seems to be about 45 per cent in favor of the plumbojarosite.

Mixtures of Minerals Ground Together: To more nearly approach conditions of practice, calcite and plumbojarosite were ground together in a pebble mill prior to flotation. One to one weight of minus 65 plus 100 mesh of calcite and plumbojarosite were ground together; one lot in a pebble mill at 70 per cent pulp density was ground for 15 minutes and another lot under the same conditions for 30 minutes. The results of grinding were as follows:

Screen analysis of 15 minute grind

Screen Size	Per cent of Total Weight.
+100 mesh	5.0
+150 "	17.5
+200 "	13.6
-200 "	68.7

Screen analysis of 30 minute grind

Screen Size	Per cent of Total Weight	%Jarosite	%Calcite
+ 100	0.0	0.0	0.0
+ 150	1.9	1.2	0.7
+ 200	8.6	5.0	4.1
- 200	89.5	43.2	45.0

It is interesting to note that the degree of grinding is about the same for calcite and for plumbojarosite in the above experiments.

Flotation of the above products of grinding demonstrated very poor separation (See Table XIV). Separation was not as good as that obtained in the case of the mixtures of sized minerals. Sodium silicate

effected no separation but seemed to lower recovery about the same for both minerals (in fact, it had a slightly greater depressing effect on the plumbojarosite). Likewise, the test using copper nitrate proved of no benefit. Copper nitrate was used on the basis of work done by Hansen[81] in which he found that it accomplised strong depression of calcite when using oleic acid as a collector.

The only difference in the case of the mixture which was ground for the longer period was a slight increase in total recovery but no better results on separation.

These results can be explained in the light of the discussion given following the first tests. Flocculation plays a major role and its non-selective action is increased by the presence of slime size material in this case. It was suggested in the previous discussion that the jarosite particles might become more or less coated with calcite or calcium salts. The opportunity for such a coating is much greater in this case, and the very close agreement in recovery for each mineral in each case would indicate that such a condition existed.

Another factor which may materially affect the equilibrium of the system is the solubility of calcium oleate.

The failure of sodium silicate to cause deflocculation or to better the efficiency of separation may be due to consumption of the reagent by the slimes present (both minerals being of the type that would readily produce much slime on grinding) or it might be due to a more or less equal

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[81] Hansen. loc. cit. p-70.

degree of depression effected on both minerals before a concentration sufficient for deflocculation is reached. This last point is strengthened by the fact that beyond very small concentrations sodium silicate has been found to strongly depress plumbojarosite (see Table V). This was also found to be true for the calcite in the work by Hansen above mentioned. This is also born out by the data represented in Figure 14, where, although the minerals are of different size, the degree of depression is uniform.

Flotation of Synthetic Mixtures of Plumbojarosite with Quartz, Using Oleic Acid

Separation on mixtures of different sized minerals: Data on the flotation of quartz alone with oleic acid was obtainable from Mr. Iverson.[82] The quartz mineral used in the separation tests with plumbojarosite was obtained from the same source. The recoveries on plumbojarosite and quartz, separately, are compared for corresponding amounts of oleic acid in Figure 17. Figure 16 shows the recoveries when a synthetic one to one mixture of quartz and plumbojarosite ore is floated with oleic acid. The points for these curves were obtained by averaging the recoveries of each mineral as obtained by screening out the different sized in the flotation concentrate of each test. The data is presented in Table XV.

Figure 16 brings out strikingly the effect of flocculation. Quartz alone responds very poorly to oleic acid as shown in Figure 17, but when

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[82] Iverson. Private communication. loc. cit.

in the presence of the flocculently floated plumbojarosite, it is very effectively entrained and buoyed up by the heavy flocs of the jarosite.

Separation of Mixtures of the same sized minerals: Separation tests were not run on mixtures of quartz and plumbojarosite ground together. It is realized that such a grinding combination would probably produce slime size of the bulk of the plumbojarosite before an appreciable reduction of quartz would result.

The above tests were not entirely satisfactory for the same reason given for the similar tests with calcite mixtures, namely, that the coarser size of jarosite was shredded and reduced appreciably during the flotation process and hence the separation based on screening was more or less in error. This difference was still greater in the case of the quartz mixtures because of the hardness of quartz. To obviate this difficulty a mixture was made of the same sizes of mineral and the respective recoveries were determined by analysis. Figure 18 shows the results of separation tests on this mixture, when using sodium silicate for a peptizing agent. Within a narrow margin, as regards concentration of sodium silicate, this reagent may be used to advantage in separation of plumbojarosite from quartz. At a concentration of 0.025 pound of sodium silicate per ton and when using 0.025 pound of oleic acid the maximum separation is obtained--100 per cent recovery of plumbojarosite and 30 per cent recovery of quartz. At greater concentrations of sodium silicate plumbojarosite is rapidly depressed. A greater efficiency than this is probably not obtainable because at this point the degree of deflocculation is probably as complete as possible to obtain a reduction in

recovery of quartz to 30 per cent and yet allow sufficient collecting action to obtain 100 per cent recovery of plumbojarosite.

The difference in action of sodium silicate on quartz-jarosite mixtures as compared with calcite-jarosite mixtures is most probably due to the fact that quartz is not floated by oleic acid and when flocculation is broken up there is no reason why quartz should persistin floating.

Flotation of Plumbojarosite with Xanthates

Introduction: The xanthates, particularly ethyl xanthate, have been important reagents in the flotation of sulphide minerals for some time.[83] More recently they have come into use,principally amyl xanthate,in the flotation of oxidized ores.[84]

The fact that higher xanthes are proving successful in practice plus the experimental varification that; " the activity of a collector from a homologous series of compounds increases as the nonpolar part of the reagent molecule increases",[85] suggested the investigation of the action of higher xanthates on plumbojarosite.

Experimental Results: Four xanthates were selected to furnish a fair representation of the straight chain series. A list of these,including the formulae and number of carbon atoms, is given accompanying Figure 19. The flotation data for these reagents is given in Table XVI and the recovery curves shown in Figure 19. Ethyl xanthate has a marked depressing effect on plumbojarosite; amyl xanthate increases the recovery from 32 per cent, (obtained with 0.5 pound terpineol per tonalone) to a max-

imum of 48 per cent at a concentration of 2.5 poundsper ton; octyl xanthate increases the recovery to 100 per cent at a concentration of alittle over 3.0 pounds per ton; and lauryl xanthate increases recovery to 100 per cent at a concentration of 1.0 pound per ton. These curves are in good agree- with the homologous series relation to flotation.

Tests No. 234 and 235 show the effect of acid and base, respectively, on the recovery with amylxanthate. Similar to all previous cases this effect is one of depression and again agrees, roughly, with the pH degree.

Tests No. 242 and 243 show the effect of slime sized material on the recovery with lauryl xanthate. Whereas this reagent gave a 100 per cent recovery on minus 200 plus 560 mesh plumbojarosite for a concentration of 1.0 pound per ton, it effected only 80 per cent recovery on minus 560 mesh mineral at a concentration of 2.0 poundsper ton (see page 56 for other data on slimes). The reason for the still lower recovery (Test No. 243) at a concentration of 3.5 pounds lauryl xanthate per ton was probably not due to any effect of the reagent or the slimes but to the effect of the high concentration of alcohol on the froth (Lauryl xanthate was dissolved in alcohol; the other three xanthates used were dissolved in water). The froth was thing and brittle and astrong oder of camphor was given off during the test.

Theoretical considerations: As in the case of many other reactions in flotation, when fundamental explanations are attempted, there seems to

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[83] Weinig and Palmer. loc. cit. p - 23

[84] Harry R. Wilson. Froth Flotation of Oxidized Lead-Silver Ore. U.S. Pat. 1,733,570., Oct. 1929.

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[85] Gaudin and Sorensen. FlotationFundamentals, Part 2. loc. cit. p-7

be considerable disagreement among authorities on the mechanism of flotation with the xanthates. Gaudin[86] in discussing the action of the xanthates on pyrite makes the following statement: "The action of xanthate as a collector may be explained in the following manner. When xanthate ionizes, the xanthate ion $(C_2H_5)OCSS^-$ may attach itself by means of its unsatisifed valence and stray valence, to the superficial atoms consitituting the pyrite grains. Thus the non-polar end of the xanthate ion becomes oriented away from the sulphide, that is, toward the water. Such a particle will adhere to an air surface because the non-polar part of the xanthate ions (the hydrocarbon chain) is oriented toward the air, and that a reduction in the surface energy of the system results from the substitution of a coated-mineral air interface for a coated-mineral solution and a solution-air interface". Taggart,[87] on the other hand, says: "All dissolved reagents which, in flotation pulps, either by action on the to-be floated or the not-to be floated particles affect their floatability, function by reason of chemical reactions of well recognized types between the reagent and the particle affected". Weiser[88] states: it seems altogether probable that the polysulphides which Gaudin assumes, areadsorption complexes rather than Dalton compounds".

Taggart[89] also states that even freshly ground galena surfaces quickly oxidize to lead sulphate or allied compounds. Further, he believes that in subsequent flotation with xanthates, the xanthate radical is abstracted

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[86]Gaudin and Orr. Flotation Fundamentals, Part 1. loc. cit. p-36.
[87]Taggart. Chemical Reactions in Flotation. loc. cit. p-5.
[88]Weiser. The Colloidal Salts. McGraw-Hill, New.York, 1928, p-146.
[89]Taggart. loc. cit. p-19

to form the insoluble lead xanthate and the sulphate radical is thrown into solution. This he backs up by tests for the sulphate radical, proving that when xanthate is added to the pulp a larger amount of sulphate radical is thrown into solution. This procedure should hold for plumbojarosite in its flotation with the xanthates if the same reason for flotation holds.

Plumbojarosite contains the sluphate radical which must be associated with etther the lead or the iron or both. To check this possibility qualitative tests were made onthe pulp of several runs using slime sizes and a one to one dilution, both with and without additions of xanthates. A check was made on the quantity of SO_4 present due to the calcium sulphate in the ore. Large quantities,(100 grams),were agitatied with water on rolls for 20 hours and the filtered solutioms analyzed quantitatively for SO_4 and Ca. In each case the SO_4 was satisfied by the Ca present. Also sensitive qualitative tests for iron and lead were negative in each case. These results indicate that the flotation of plumbojarosite with xanthate is due to an adsorption (which may be caused by a "residual" or "stray" valence or some such expression of surface energy or electrical charge) rather than a simple chemical reaction.

Experimental Work on Tintic Standard Ore

Considerable preliminary work has been done on this ore. None of the data obtained is presented in this paper however, because the investigation has not been carried to the point where definite conclusions can be made. Also, it has been decided to make an extended program of this phase of the work and include in it. the development, if possible, of a differential method of chemical analysis for argentojarosite. Obviously, argento-

jarosite has the greatest ecomonic significance whenever these minerals are present in appreciable amounts.

The work so far has indicated that what has been presented in this paper on the flotation of plumbojarosite has valuble application to the development of a practical problem such as is presented by this Tintic Standard ore. So far, flotation with the higher xanthates seems the most favorable possibility.

CONCLUSIONS

Plumbojarosite and argentojarosite are more common minerals than is generally realized. Their presence is an important factor in metal losses in oxidized ore flotation because of their peculiar characteristics such as: unresponsiveness to recovery by the reagents ordinarily used in oxidized ore flotation; tendency to occur in, or crush readily to, slime sizes; low specific gravity; complex structure; similarity to gangue minerals such as iron oxides.

In general the best flotation recoveries of plumbojarosite are made from a neutral or slightly acidic circuit. Any considerable degree of variation from neutral in either direction causes depression.

Plumbojarosite does not sulphidize when treated in solutions of strengths employed in practice and for periods ordinarily used. Sodium sulphide blackens the mineral after 3 hours of contact but the result is detrimental to the flotation of the same.

Plumbojarosite does not sulphidize with hydrogen sulphide gas, although it is blackened after 1 hour of treatment. The blackening is due to an accumulation of sulphur at the boundaries of the groups of platy crystals. The result was depression for all subsequent collectors tried, except oleic acid.

Plumbojarosite floats readily with sulphureted pine oil. The action is not sulphidizing.

Plumbojarosite is floated very readily by the fatty acids of

both the saturated and the unsaturated series. Increase in temperature of the pulp increases the recovery with the fatty acids of higher melting points.

Slime sizes of plumbojarosite consume over ten-fold the quantity of fatty acid necessary to obtain corresponding recovery on minus 200 plus 560 mesh plumbojarosite.

The fatty acids are not sufficiently selective in their flotation action. No efficient separation of jarosite can be made from mixtures with calcite. By close regulation of deflocculating agent a fair degree of separation may be made from mixtures with quartz.

The higher xanthates are good collectors for plumbojarosite.

Since plumbojarosite contains a high percentage of iron any reagent used for its recovery must be such that it makes a clean recovery of the plumbojarosite without excessive dilution of the concentrate with gangue. The margin is not so close on argentojarosite which is a high grade silver mineral.

Experimentation with a typical oxidized jarosite-bearing ore indicates that the use of higher xanthates may bring about a good recovery of plumbojarosite (as well as other values) in the relatively fine sizes, but that the true slimes present a difficult problem.

The most important phases of the problem for further investigation are: the chemical and physical structure of plumbojarosite;

a differential chemical method of analysis for argentojarosite; an investigation of the electrical relations in the flotation cell; flotation with the higher xanthates applied to slimes and to separation possibilities in synthetic mixtures with gangue minerals and in actual ores.

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TABLES AND PLATES

TABLE III

Flotation of plumbojarosite using varying concentrations of terpineol expressed in pounds per ton of ore. Recovery expressed in per cent.

Test [a] No.	Terpineol	pH	Recovery
	Minus 200 plus 560 mesh plumbojarosite		
1	0.0	7.1	7.2
2	0.1	7.1	14.4
3	0.5	7.1	32.0
4	1.0	7.0	56.0
5	2.0	6.8	64.0
	Minus 150 plus 200 mesh plumbojarosite		
6	0.0	7.1	0.9
7	0.5	6.9	1.6
8	2.0	6.6	0.8
	Minus 100 plus 150 mesh plumbojarosite		
9	0.0	7.1	0.8
10	0.5	7.1	3.2
11	5.0	6.5	2.4
	Minus 65 plus 100 mesh plumbojarosite		
12	2.0	6.6	0.8
	Minus 200 plus 280 mesh plumbojarosite		
13	0.0	7.1	5.1
14	0.1	7.1	9.0
15	0.5	6.9	34.0
16	1.0	6.9	46.0
	Minus 560 ("slime") mesh plumbojarosite		
17	0.0	7.3	17.0
18	0.5	7.3	48.4
19	2.0	7.0	64.0

[a] In this and all following tables of flotation data the test numbers have been arranged in sequence for ease of reference. Nearly all tests have been run in duplicate or until properly checked, although only the average or checked values are given.

TABLE IV

Effect of various concentrations of different acids and bases on the flotation of minus 200 plus 560 plumbojarosite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery in per cent.

Test No.	Reagents	pH	Recovery
	NaOH		
20	0.1	7.2	16.4
21	0.5	7.6	11.6
22	1.0	8.9	7.2
23	2.0	10.3	5.6
	HCl		
24	0.1	6.8	37.2
25	0.5	6.5	26.8
26	1.0	5.9	16.8
27	2.0	4.3	9.2
	Na_2CO_3		
28	0.1	7.1	42.0
29	0.5	7.3	32.0
30	1.0	7.5	18.8
31	2.0	8.6	8.8
	NH_4OH		
32	0.1	7.2	40.0
33	0.5	7.4	29.2
34	1.0	8.2	17.2
35	2.0	9.2	10.8
	CaO		
36	0.1	7.2	34.4
37	0.5	7.6	12.0
38	1.0	9.9	6.8
39	2.0	9.9	8.0
	H_2SO_4		
40	0.1	7.0	17.2
41	0.5	6.9	16.8
42	1.0	6.0	8.4
43	2.0	3.6	6.0

TABLE V

Flotation of minus 200 plus 560 mesh plumbojarosite for preliminary data on reagents subsequently used in combination with sulphidizing agents, (Table VI). All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery in per cent.

Test No.	Reagents	pH	Recovery
	$(NH_4)_2CO_3$		
44	0.1	7.0	25.2
45	0.5	7.5	16.8
46	1.0	7.6	16.0
47	2.0	7.6	14.4
	$FeCl_3$		
48	0.1	7.2	22.4
49	0.5	7.0	6.0
50	1.0	6.7	4.8
51	2.0	5.8	3.6
	Na_2SiO_3		
52	0.1	7.3	32.0
53	0.5	7.5	6.0
54	1.0	7.9	4.0
55	2.0	8.1	5.6

TABLE VI

Effect of various concentrations of different sulphidizing reagents, under different conditions, on the flotation of minus 200 plus 560 mesh plumbojarosite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton of ore. Recovery in per cent.

Test No.	Sulphidizing Reagent	Other Reagents		Preagitation Time, minutes	pH	Recovery
	Na_2S					
56	0.1	0.0		4	7.1	25.2
57	0.5	0.0		4	7.3	818
58	1.0	0.0		4	7.5	6.8
59	2.0	0.0		4	8.5	4.0
60	0.5	0.0		8	7.3	10.8
61	0.5	0.0		12	7.3	9.2
62	0.5	0.0		60	7.2	3.0
		$(NH_4)_2CO_3$				
63	0.1	0.5		4	7.1	22.4
64	0.5	0.5		4	7.6	11.2
65	1.0	0.5		4	7.9	8.8
66	2.0	0.5		4	8.3	8.8
		$(NH_4)_2CO_3$	K.E.X.			
67	30.0	0.0	1.0	60.	8.0	2.4
68[b]	0.5	0.5	0.1	4	7.5	9.2
69	0.5	0.5	0.5	4	7.5	818
70	0.5	0.5	1.0	4	7.6	10.4
71	0.5	0.5	2.0	4	7.6	12.3
72	0.5	0.5	3.0	4	7.6	6.4
73	2.0	0.5	0.0	60	7.7	8.4
74	2.0	0.5	0.0	12	7.2	7.2
75	1.0	1.0	1.0	30	7.6	12.3
		Na_2SiO_3				
76[c]	5.0	5.0		4	8.8	6.0
		$FeCl_3$-brine				
77	1.0	25.0 cc.[d]		2	3.5	3.2
78	5.0	25.0 cc.		8	2.6	4.0

TABLE VI
(Continued)

Test No.	Sulphidizing Reagent	Other Reagents	Preagitation Time, minutes	pH	Recovery
	H_2S	$(NH_4)_2CO_3$			
79	0.1	0.5	4 min. for	7.5	13.2
80	0.5	0.5	each rea-	7.9	12.8
81	1.0	0.5	gent.	7.5	10.0
82	2.0	0.5		7.3	8.0
		Cu_2SO_4			
83	0.5	0.5	Cu_2SO_4 - 2 H_2S - 4	6.5	8.4
	H_2S [e]	K.A.X.			
84		2.5	4	6.4	32.0
84A	(on tailings of 84)	10.0	4		0.0
		K.E.X.			
85		2.5	4	6.5	15.4
		Cu_2SO_4			
85A	(on tailings of 85)	3.0	4	6.4	14.0
	$(NH_4)_2S_5$ [f]	$FeCl_3$			
86	1.0	none	4	9.6	3.6
87	0.1	0.1	4	9.7	2.8
88	5.0	5.0	4	9.7	3.6
		$FeCl_3$-brine			
89	5.0	25.0cc	Brine - 60 $(NH_4)_2S_5$ - 4	7.1	6.4
	Sulphureted Pine Oil				
90	0.368	none	4	6.5	100.0
91	0.184	none	4	6.5	86.7

TABLE VI
(Continued)

Test No.	Sulphidizing Reagent	Other Reagents	Preagitation Time, minutes	pH	Recovery
	Sulphureted Pine Oil	HCl			
92	0.368	0.5	4	3.0	10.0
93	0.368	1.0	4	2.3	4.0
		NaOH			
94	0.368	1.0	4	7.5	63.1
95	0.368	none	4	6.7	100.0

[b] In tests No. 68 to 72 inclusive the preagitation time was 2 minutes after addition of $(NH_4)_2CO_3$, then addition of Na_2S and 4 minutes more time, then addition of K.E.X. (potassium ethyl xanthate) and 2 minutes more agitation. In tests No. 73 the preagitation time was 2 minutes with the $(NH_4)_2CO_3$ followed by 60 minutes with Na_2S. In test No. 74 the preagitation time was 12 minutes with the $(NH_4)_2CO_3$ followed by 12 minutes with the Na_2S. In test NO. 75 the preagitation time was 10 minutes with the $(NH_4)_2CO_3$ followed by 30 minutes with the Na2S followed by 4 minutes with the K.E.X.

[c] Ferric chloride-brine solution. This was a solution of the same strength as is used in the analytical method for plumbojarosite (see page 22). The strength, calculated in pounds of contained salts per ton of ore, is 4 pounds $FeCl_3$ per ton per cc. of solution and 28.8 pounds of NaCl per ton per cc. of solution.

[d] The ore used for tests No. 84 and 85 was treated with H_2S by agitating a charge of the mineral in an aqueous saturated solution for 4 hours.

[e] $(NH_4)_2S_5$. Ammonium pentasulphide; the concentrations given for this reagent are expressed in pounds of sulphur per ton of ore.

[f] Test No. 95 was run in an aluminium paint coated iron cell instead of the celluloid cell.

TABLE VII

Effect of various periods of treatment with dry hydrogen sulphide gas on the flotation of minus 200 plus 560 mesh plumbojarosite, under different condition, All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Preagitation time in minutes. Recovery in per cent. Sulphidizing time in minutes.

Test No.	Sulphidizing Time	Other Reagents	Preagitation Time	pH	Recovery
	H_2S gas				
96	30	none	4	6.3	15.2
97	120	none	4	6.0	4.0
		K.A.X.			
98	30	2.5	4	6.6	41.2
99	90	2.5	4	6.5	19.2
		Oleic Acid			
100	90	0.1	4	6.5	100.0
101	90	0.05	4	6.5	100.0
		Thiocarbanilid			
102	90	1.0	4	5.8	9.6
		Sulphureted terpintine derivative[g]			
103	none	0.180	4	6.5	85.6
104	240	0.180	4	6.5	5.2
		Sulphureted Pine Oil[h]			
105	none	0.184	4	6.5	94.0
106	240	0.184	4	6.5	5.6
107	120	0.368	4	6.5	2.8

[g] Sulphureted terpintine derivative; prepared by Mr. Kidd in the departmental laboratory by distilling a terpintine derivative with sulphur.

[h] Sulphureted pine oil; perpared by the author by distilling pine oil with sulphur in a reflux still.

TABLE VIII

Effect of sodium oleate on the flotation of plumbojarosite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton of ore. Recovery in percent.

Test No.	Sodium Oleate	Other Reagents	pH	Recovery
	Minus 200 plus 560 mesh plumbojarosite			
108[1]	0.05		7.0	13.2
109	1.0		7.1	100.0
110	0.5		7.1	100.0
111	0.2		6.5	100.0
112	0.1		6.9	98.0
113	0.05		7.1	98.0
114	0.025		7.1	97.5
115	0.01		6.8	84.0
		HCl		
116	0.05	0.1	6.9	97.5
117	0.05	1.0	6.6	81.5
118	0.05	2.0	4.6	34.0
		NaOH		
119	0.05	1.0	9.7	62.8
		$NaHCO_3$		
120	0.05	1.0	7.4	77.2
		$Pb(NO)_3$		
121	0.05	0.05	6.9	100.0
	Minus 150 plus 200 mesh plumbojarosite			
122	0.5		6.8	100.0
123	0.0		6.9	3.2
124	0.01		7.0	3.6
125	0.025		6.7	11.6
126	0.04		6.7	47.2
127	0.05		6.7	96.0
128	0.075		6.9	100.0
129	0.1		6.9	100.0

[1] Test No. 108; no frother used

TABLE IX

Effect of oleic acid on the flotation of plumbojarosite. All reagents in pounds perton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery in per cent.

Test No.	Oleic Acid	Other Reagents	pH	Recovery
	Minus 200 plus 560 mesh plumbojarosite			
130	0.5		6.5	98.0
131	0.1		6.5	100.0
132	0.05		6.5	100.0
133	0.025		6.5	86.4
134	0.01		6.5	72.8
135	2.0			96.8
	Minus 150 plus 200 mesh plumbojarosite			
136	0.075		6.5	100.0
137	0.05		6.5	96.0
138	0.025		6.5	79.1
	Minus 100 plus 150 mesh plumbojarosite			
139	0.075		6.7	70.0
140	0.1		6.7	100.0
141	0.025		6.5	6.4
142[j]	0.075		6.5	82.6
143	0.05		6.7	39.6
	Minus 200 plus 560 mesh plumbojarosite			
		Na_2CO_3		
144	0.01	1.0	7.6	64.4
		H_2SO_4		
145	0.01	0.0	6.9	57.2
146	0.01	1.0	5.9	24.8

[j] Test No. 142; mineral was washed very thoroughly with water previous to test.

TABLE IX
(Continued)

Test No.	Oleic Acid	Other Reagents	pH	Recovery
	Minus 560 mesh (slime size) plumbojarosite[k]			
147	0.0		7.3	48.4
148	0.01		7.2	55.2
149	0.05		7.5	63.2
150	0.5		6.9	100.0
		H_2SO_4		
151	0.05	1.0	6.9	59.0
		Na_2CO_3		
152	0.05	1.0	7.9	60.4
	Minus 560 mesh plumbojarosite from La Plata			
153	0.0		7.9	44.8
154	0.2		7.7	82.8

[k] See Table XVI for effect of xanthates on slimes.

TABLE X

Alcohol leaches on minus 200 plus 560 mesh plumbojarosite for removal of gypsum. Effect of oleic acid on plumbojarosite after leaches. Oleic acid in poundsper ton. Constant amount of terpineol, o.5 pound per ton. Recovery in per cent. Calcium content reported as CaO.

Heads . . . . Mexican plumbojarosite . . . . . 0.36% CaO

Leach No.	Time	% CaO remaining
1	18 hrs.	0.25
2	36 hrs.	0.12
3	54 hrs.	0.0

Flotation

Test No.	Oleic Acid	Leach No.	pH	Recovery
161	0.0	1	6.5	37.6
162	0.01	2	6.5	82.0
163	0.0	3	6.5	48.0
164	0.01	3	6.5	80.1

TABLE XI

Effect of various concentrations of saturated and unsaturated fatty acids on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound of terpineol per ton of ore at room temperature. Recovery in per cent.

Test No.	Fatty Acid	pH	Recovery
	Crotonic		
165	0.1	6.5	41.2
166	0.5	5.6	48.4
167	1.0	4.9	28.0
	Undecylenic		
168	0.1	6.5	100.0
169	0.05	6.9	95.0
170	0.01	6.5	52.0
	Oleic [1]		
	Heptylic		
171	0.01	6.5	33.1
172	0.05	6.5	43.2
173	0.10	6.5	64.2
174	0.35	6.5	78.4
175	0.5	6.5	85.0
176	2.0	4.5	98.0
	Lauric		
177	0.1	6.3	100.0
178	0.02	6.3	100.0
179	0.01	6.3	96.7
180	2.0	5.8	90.0
	Stearic		
181	0.05	6.5	45.6
182	0.1	6.5	64.0
183	0.2	6.5	90.0
184	0.35	6.5	96.0
185	0.5	6.5	100.0

[1] Oleic acid; see Table IX

TABLE XII

Effect of various concentrations of fatty acids on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore and, of terpineol alone when maintaining pulp temperatures above melting point of the fatty acid. Reagents in pounds per ton of ore. Recovery in per cent.

Test No.	Fatty Acid	Temperature in °C.	pH	Recovery
	Crotonic			
186	0.0	75 to 65	--	85.2
	Lauric			
187	0.005	50	--	98.4
188[m]	0.005	50	6.5	100.0
	Stearic			
189	0.2	50	6.7	100.0
190	0.01	70 - 62	6.5	100.0
	Terpineol only			
191	0.5	70 - 60	6.5	67.6
192[n]	015	70 - 60	6.5	44.6
193[o]	0.5	70 - 60	6.5	100.0
	No Terpineol			
194	0	70 - 60	6.5	22.0

[m] Test No. 188 run in iron cell (aluminium paint-coated) with propeller shaft uncoated.

[n] Test No. 192 run incelluloid cell with propeller shaft uncoated.

[o] Test No. 193 run in iron cell (aluminium paint coated) with propeller shaft uncoated.

TABLE XIII

Effect of oleic acid and sodium silicate on the flotation of minus 100 plus 150 mesh calcite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery in per cent.

Test No.	Oleic Acid	Sodium Silicate	pH	Recovery
195	none		8.7	1.6
196	0.05		8.6	26.8
197	0.075		8.6	34.8
198	0.1		8.3	48.0
199	0.2		8.1	70.4
200	0.5		7.9	100.0
201	0.075	0.1	8.6	6.8
202	0.075	1.0	9.6	1.2
203	0.075	0.2	9.1	1.2

TABLE XIV

Effect of oleic acid and sodium silicate on the differential flotation of one to one mixtures of calcite and plumbojarosite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery in per cent.

Test No.	Oleic Acid	Sodium Silicate	pH	Jarosite Recovery	Calcite Recovery
	Minus 100 plus 150 mesh calcite and minus 200 plus 560 mesh plumbojarosite.				
204	0.075	0.2	8.7	5.6	1.6
205	0.075	0.1	8.6	36.0	1.6
206	0.075	0.0	8.5	94.4	48.0
	Minus 200 mesh calcite and minus 100 plus 150 mesh plumbojarosite				
207	0.075	0.0	8.0	2.4	94.5
	Minus 100 plus 150 mesh calcite and minus 100 plus 150 mesh plumbojarosite				
208	0.075	0.0	8.0	80.7	20.0
	Calcite and plumbojarosite ground together [P]				
209	0.025	0.0	7.5	39.2	36.8
210	0.05	0.0	7.3	51.2	48.0
211	0.05	0.05	7.4	48.8	48.8
212	0.05	0.0	7.9	47.2	52.8
213	0.05	0.025	7.9	35.2	56.0
		$Cu(NO_3)_2$			
214	0.05	1.0	5.9	18.4	22.4

[P] See page 67 for time of girnd and screen analyses.

TABLE XV

Effect of various concentrations of oleic acid and sodium silicate on the differential flotation of synthetic one to one mixtures of quartz and plumbojarosite. All reagents in pounds per ton of ore. Constant amount of terpineol, 0.5 pound per ton. Recovery on per cent.

Test No.	Oleic Acid	Sodium Silicate	pH	Jarosite Recovery	Quartz Recovery
214 [q]	0.005		6.7	4.0	16.0
215 [r]	0.005		6.7	64.8	8.1
216	0.025		6.5	6.4	25.6
217	0.025		6.7	93.6	92.0
218	0.0125		6.6	20.0	76.0
219	0.0125		6.5	93.6	11.7
220	0.0125	0.05	6.5	56.2	0.8
221	0.0125	0.01	6.6	93.6	7.2
222 [s]	0.025	0.0	6.5	99.2	80.0
223	0.025	0.01	6.6	99.2	31.0
224	0.025	0.02	6.7	99.2	35.2
225	0.025	0.025	6.7	99.2	32.8
226	0.025	0.25	7.0	0.8	0.8
227	0.025	0.05	6.6	74.8	25.3

[q] Tests No. 214, 216, 218, 220 were run on synthetic one to one mixtures of minus 200 plus 560 mesh quartz with minus 100 plus 150 mesh plumbojarosite.

[r] Tests No. 215, 217, 219, 221 wererun on synthetic mixtures reversed in mesh sizes to that above.

[s] Tests No. 222 to No. 227, inclusive, were run on a synthetic one to one mixture in which both quartz and plumbojarosite were minus 200 plus 560 mesh.

TABLE XVI

Effect of various concentrations of some of the xanthates on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore. Xanthates in pounds per ton of ore. Recovery in per cent.

Test No.	Xanthate	Other Reagents	pH	Recovery
	Ethyl			
228	2.5		6.7	4.0
229	015		6.5	12.0
230	10.0		7.1	6.4
	Amyl			
231	2.5		6.9	48.4
232	0.5		6.9	38.8
233	10.0		7.3	40.0
		H_2SO_4		
234	2.5	1.0	5.1	28.4
		Na_2CO_3		
235	2.5	1.0	9.6	11.2
	Octyl			
236	0.5		6.9	38.0
237	2.5		7.2	90.4
238	10.0		7.2	98.0
	Lauryl			
239	0.5		7.0	79.2
240	1.0		7.0	98.0
241	10.0		7.0	100.0
	Minus 560 mesh plumbojarosite			
	Lauryl			
242	2.0		7.7	80.0
243	3.5		7.9	68.0

PLATES

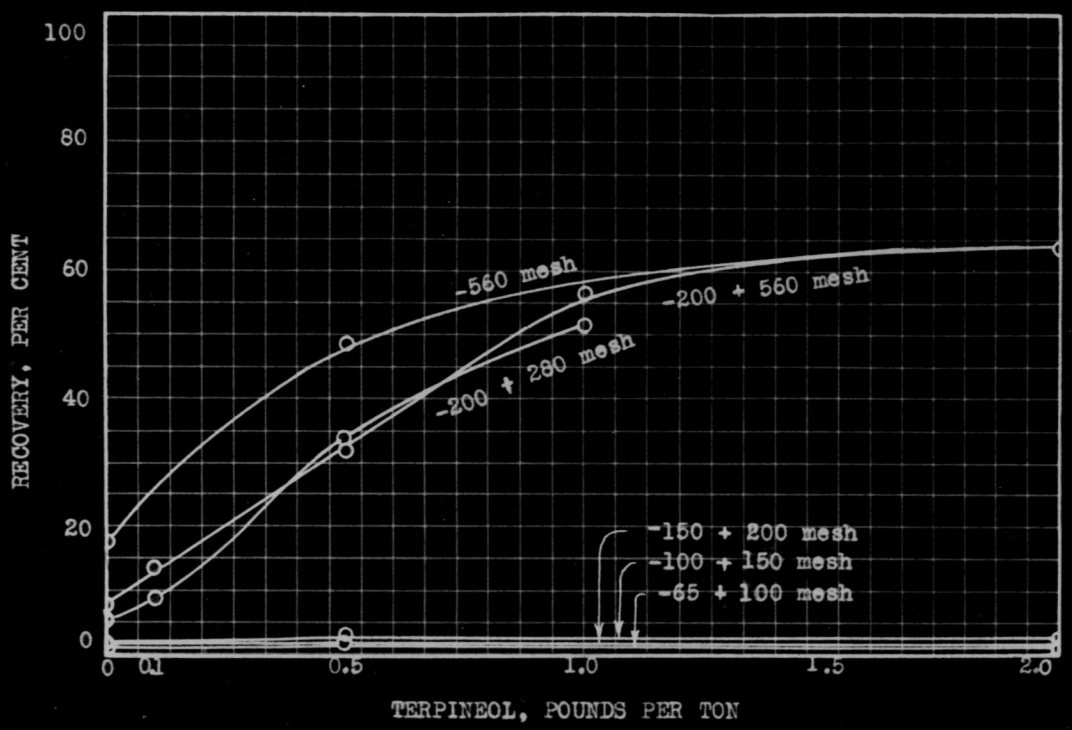

Fig. 1. Flotation of plumbojarosite using varying amounts of terpineol.

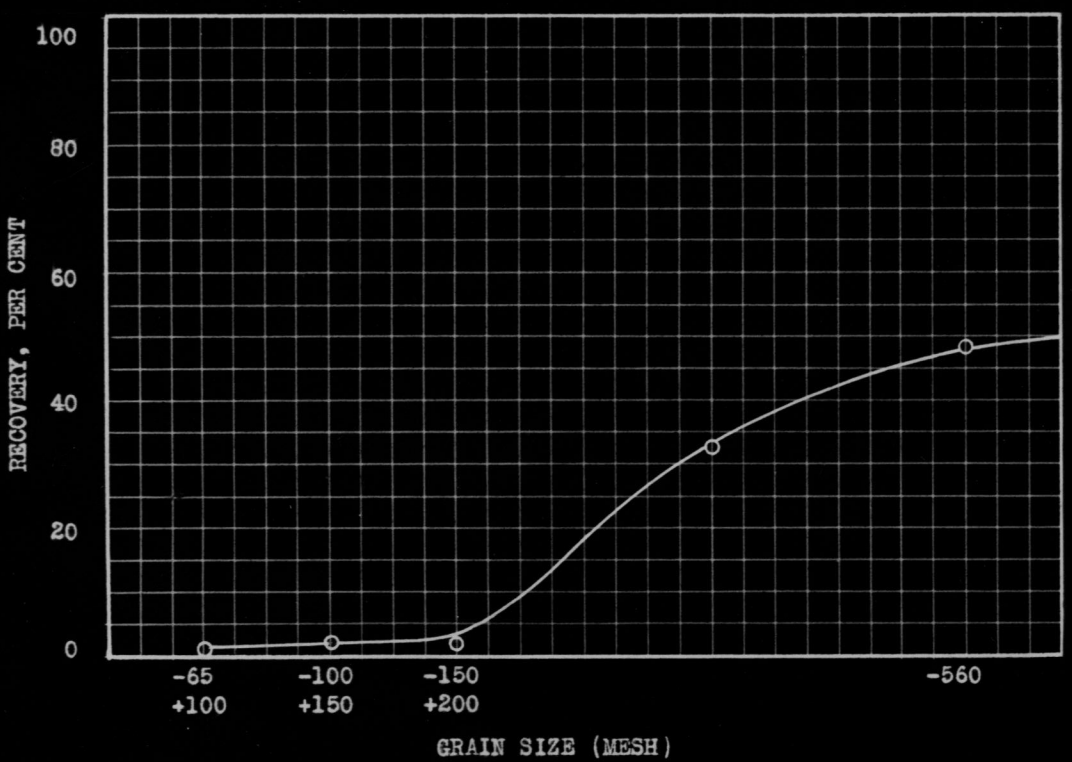

Fig. 2. Effect of grain size on flotation of plumbojarosite with 0.5 pound terpineol per ton ore.

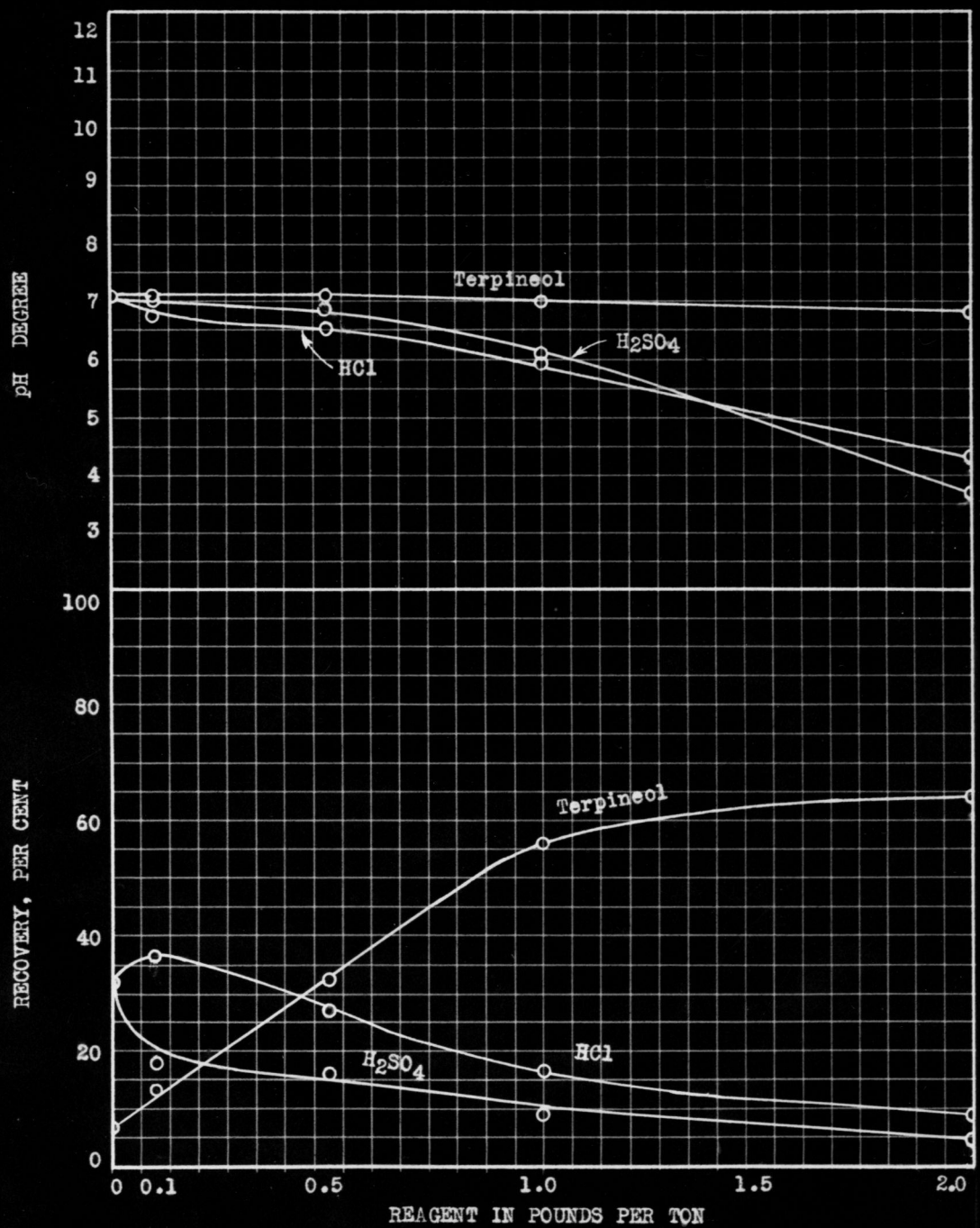

Fig. 3. Effect of various concentrations of HCl and H_2SO_4 on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore; and relation between pH degree and recovery for corresponding concentrations of acids.

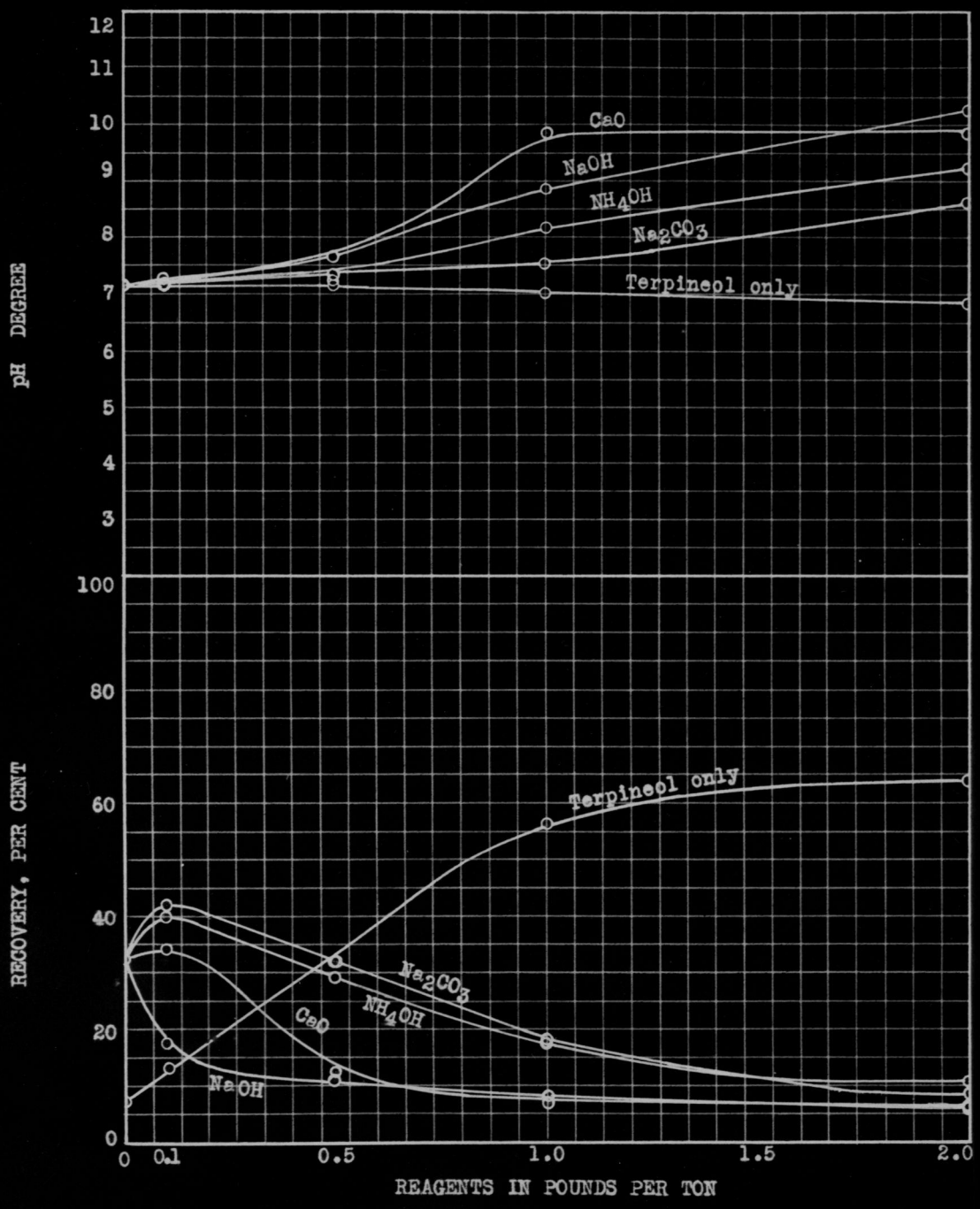

Fig. 4. Effect of various concentrations of different bases on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton.

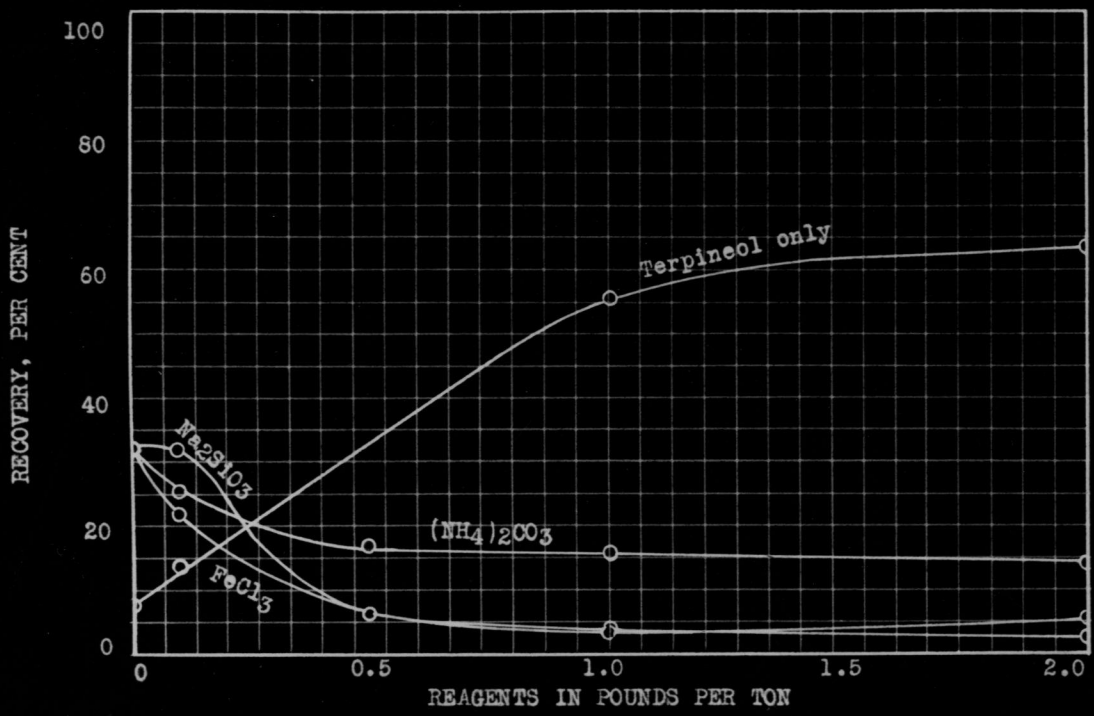

Fig. 5. Effect of various concentrations of some inorganic salts (subse - quently used in combination with sulphidizing reagents) on flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton.

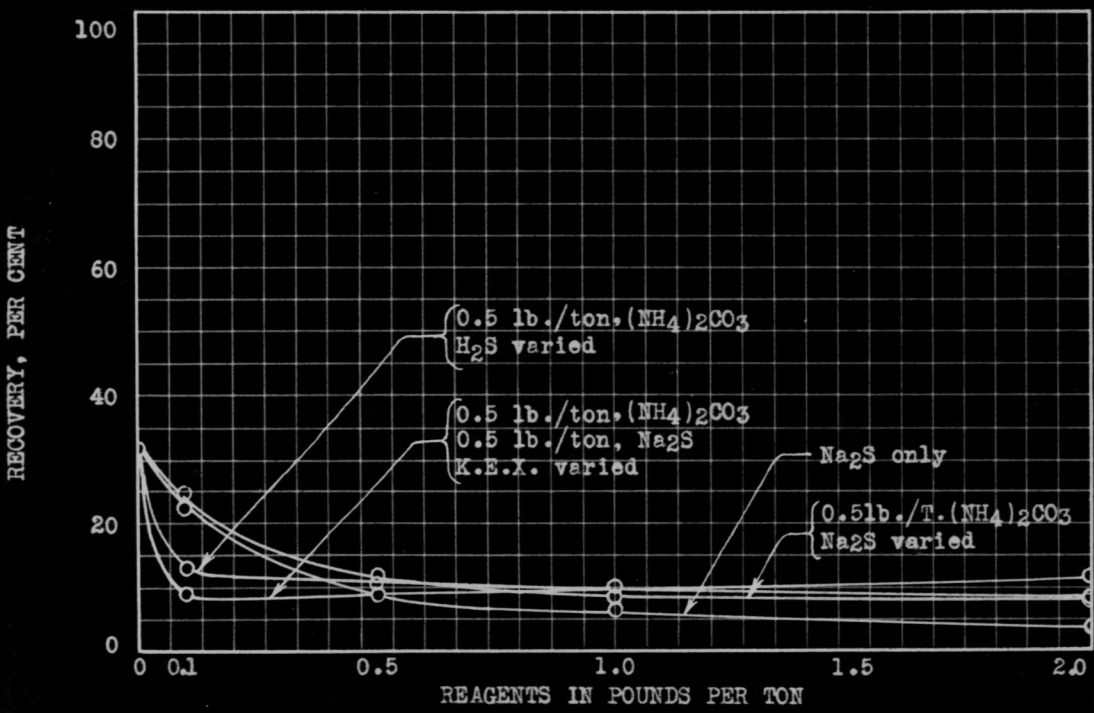

Fig. 6. Effect of various concentrations of different sulphidizing reagents, under different conditions, on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore.

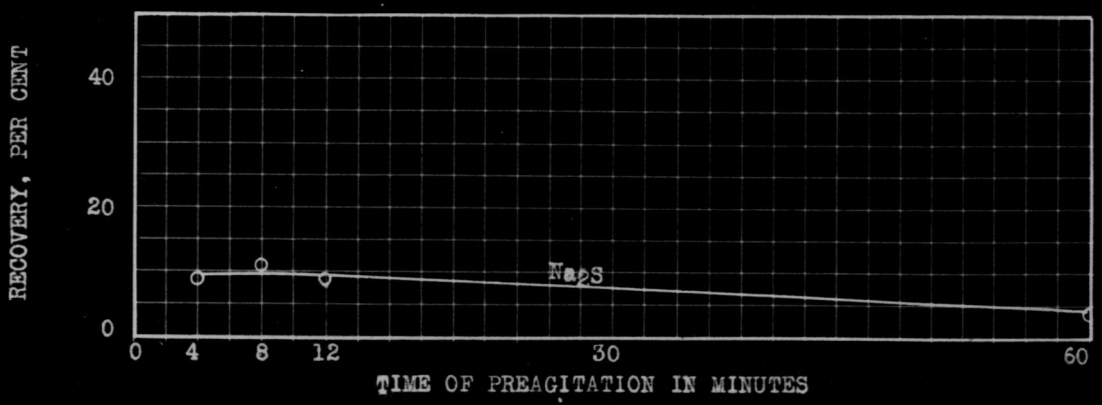

Fig. 7. Effect of time of preagitation with Na_2S on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore.

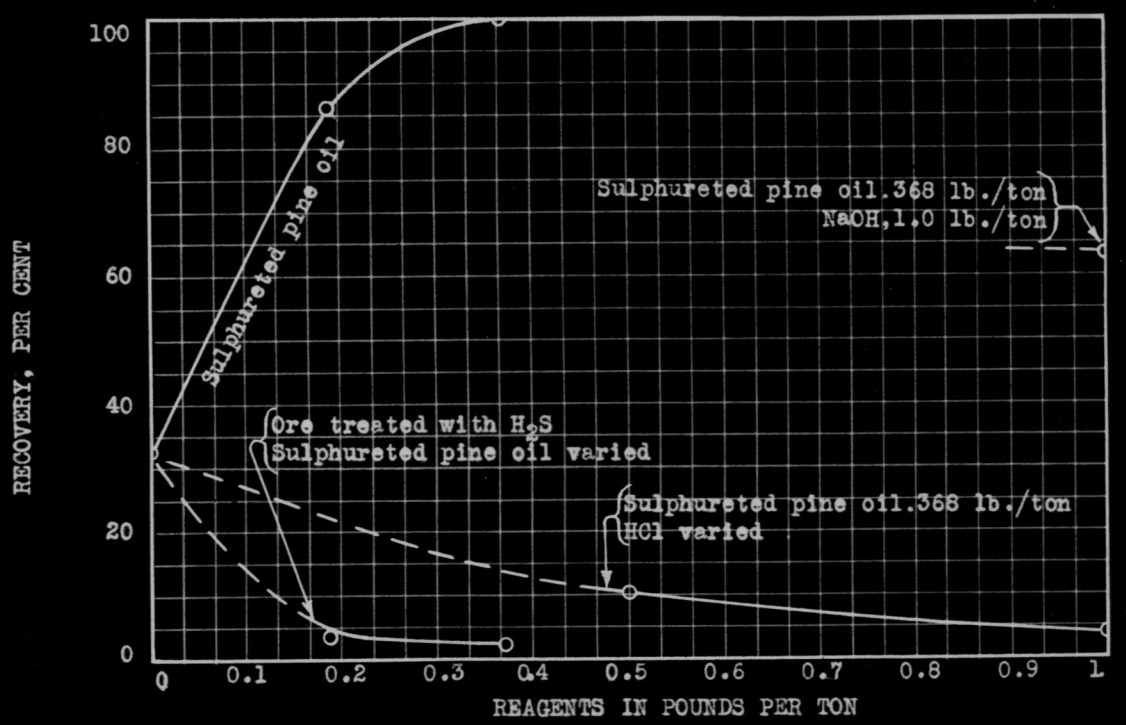

Fig. 8. Effect of various concentrations of sulphureted pine oil, under different conditions, on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore.

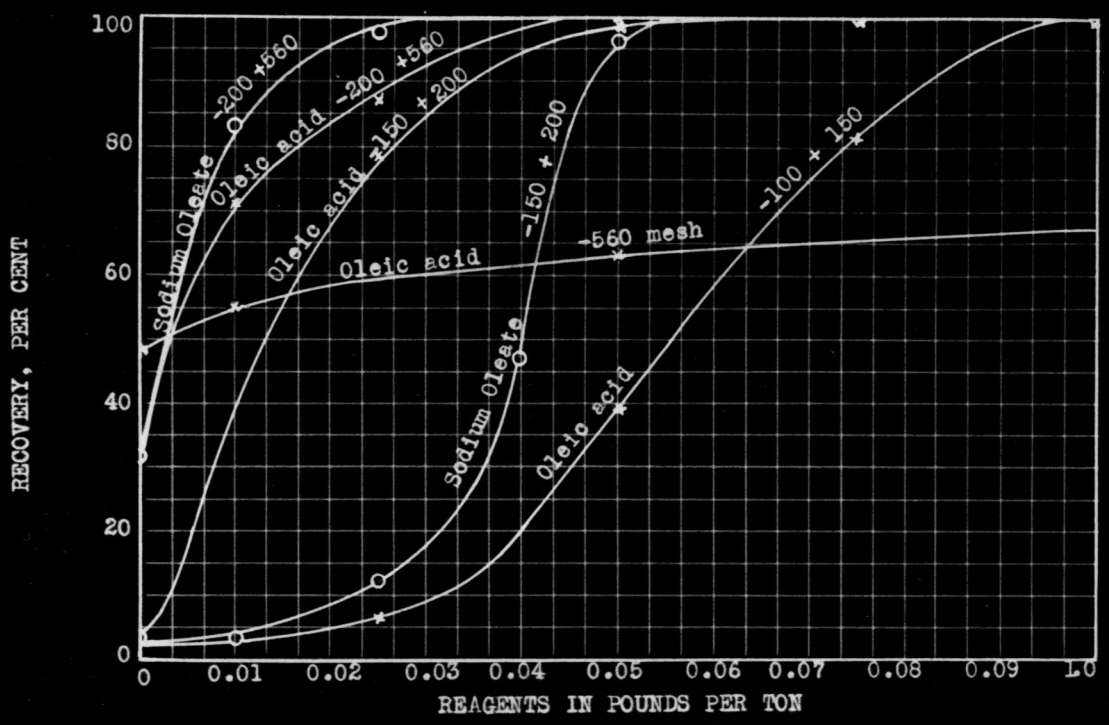

Fig. 9. Comparison of recoveries by sodium oleate and oleic acid on various sizes of plumbojarosite with 0.5 pound terpineol per ton.

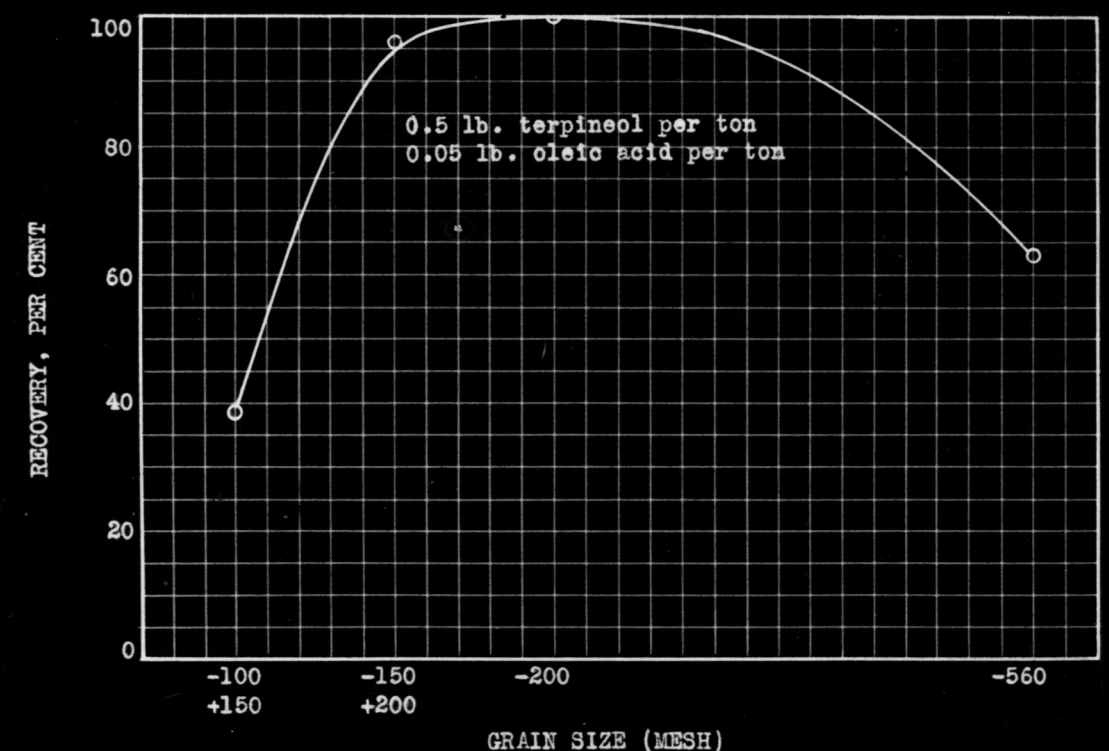

Fig. 10. Effect of grain size on flotation of plumbojarosite with 0.5 pound terpineol and 0.05 pound oleic acid per ton of ore.

FATTY ACIDS USED

SATURATED

Name	Formula	No. carbon atoms
Heptylic	$C_6H_{13}COOH$	6
Lauric	$C_{11}H_{23}COOH$	11
Stearic	$C_{17}H_{35}COOH$	17

UNSATURATED

Name	Formula	NO. carbon atoms
Crotonic	C_3H_5COOH	3
Undecylenic	$C_{10}H_{19}COOH$	10
Oleic	$C_{17}H_{33}COOH$	17

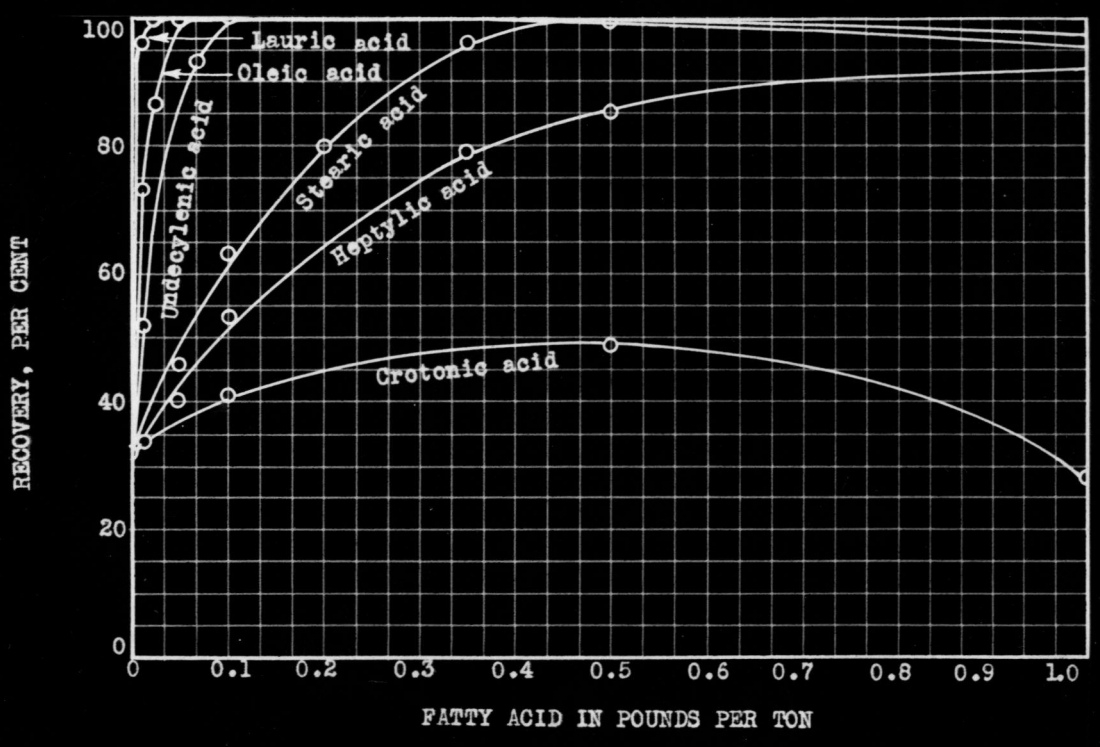

Fig. 11. Effect of saturated and unsaturated fatty acids on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore at room temperature.

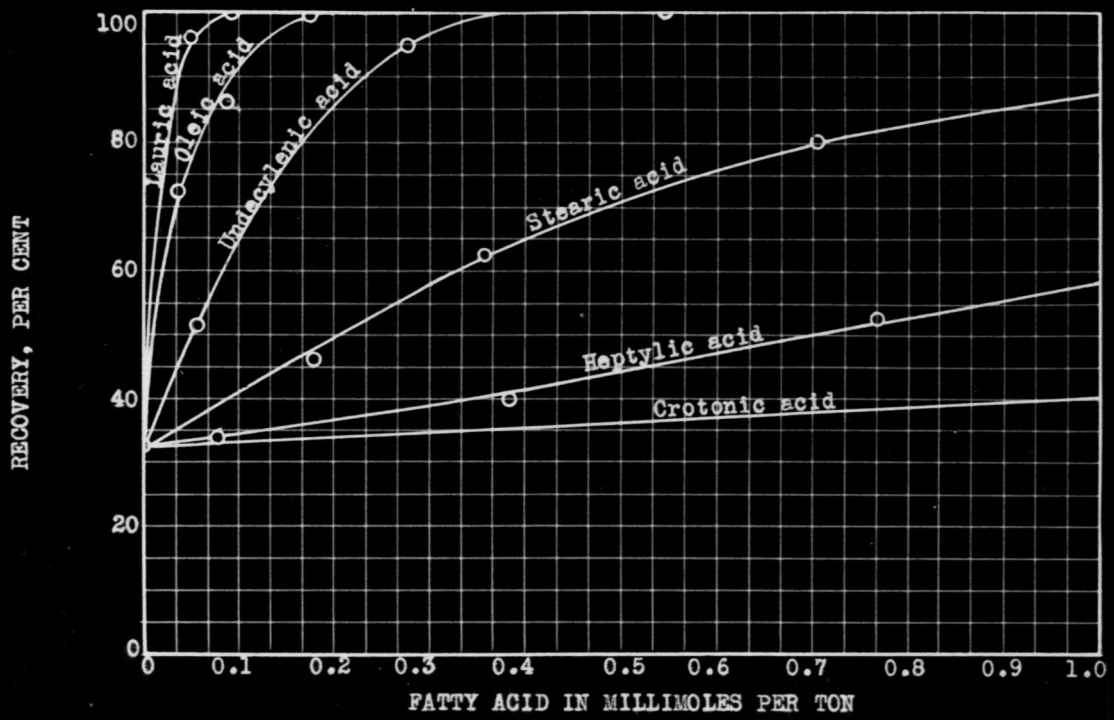

Fig. 12. Effect of saturated and unsaturated fatty acids on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore at room temperature. Concentration of fatty acids in millimoles per ton.

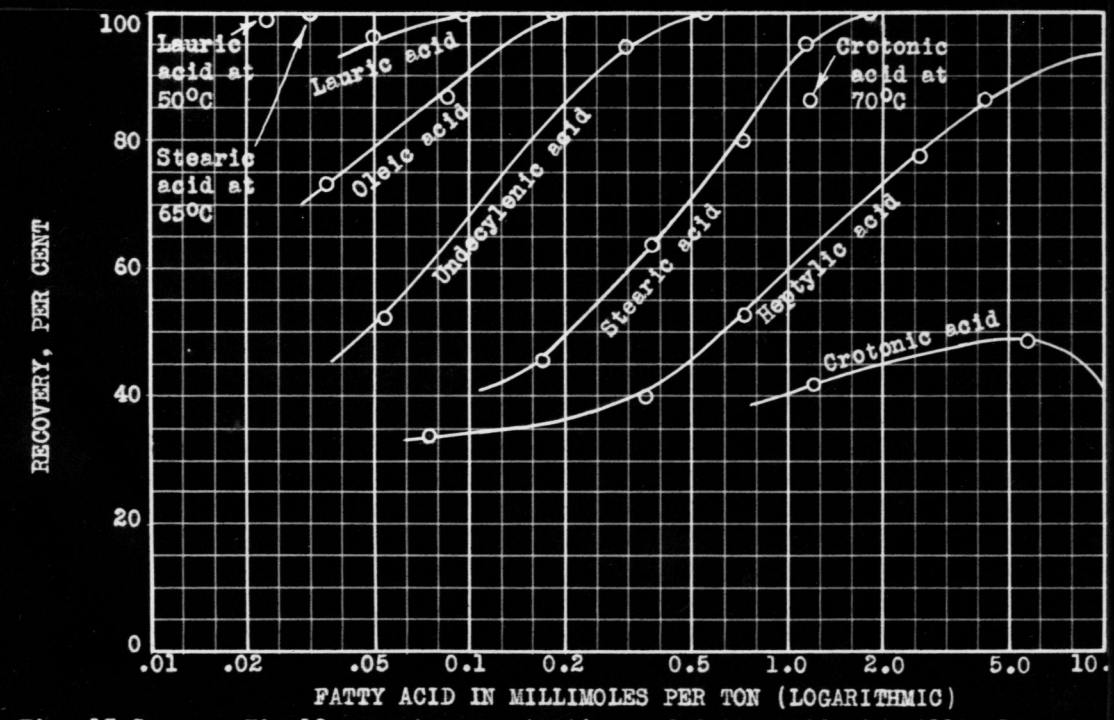

Fig. 13 Same as Fig.12 except concentrations of fatty acids in millimoles are plotted logarithmically. Also comparison of recoveries between flotation experiments conducted at room temperatures and at the respective melting points of three of the fatty acids, (crotonic, lauric, and stearic).

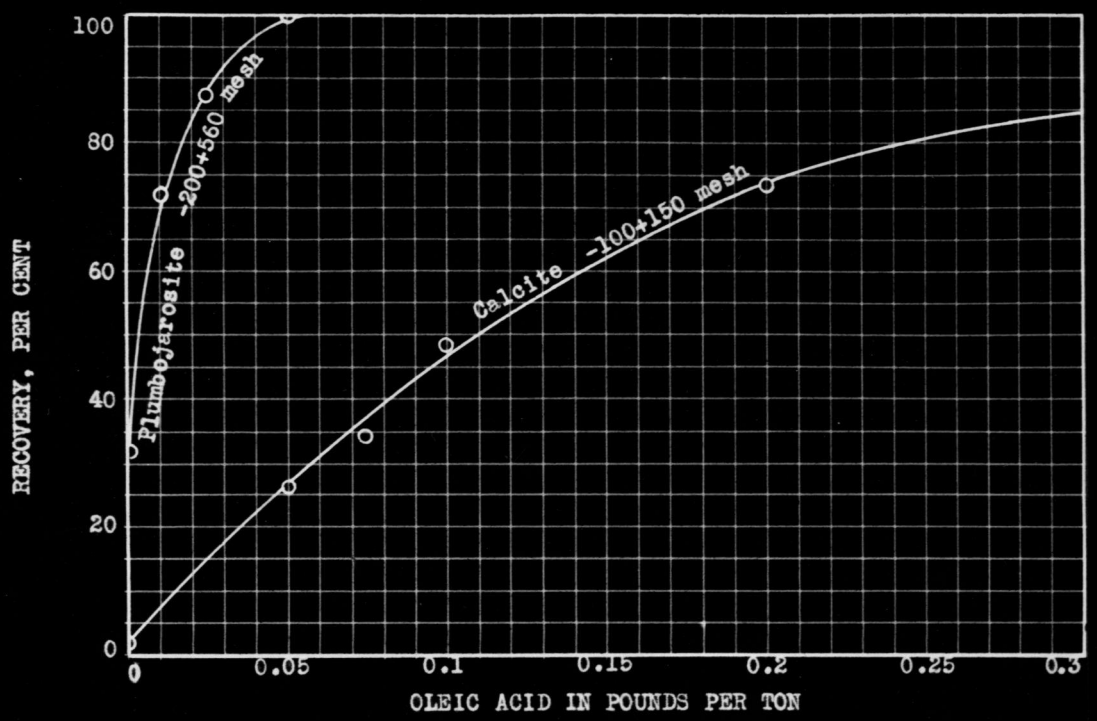

Fig. 14. Flotation of pure plumbojarosite compared with flotation of pure calcite using various concentrations of oleic acid with 0.5 pound terpineol per ton of ore.

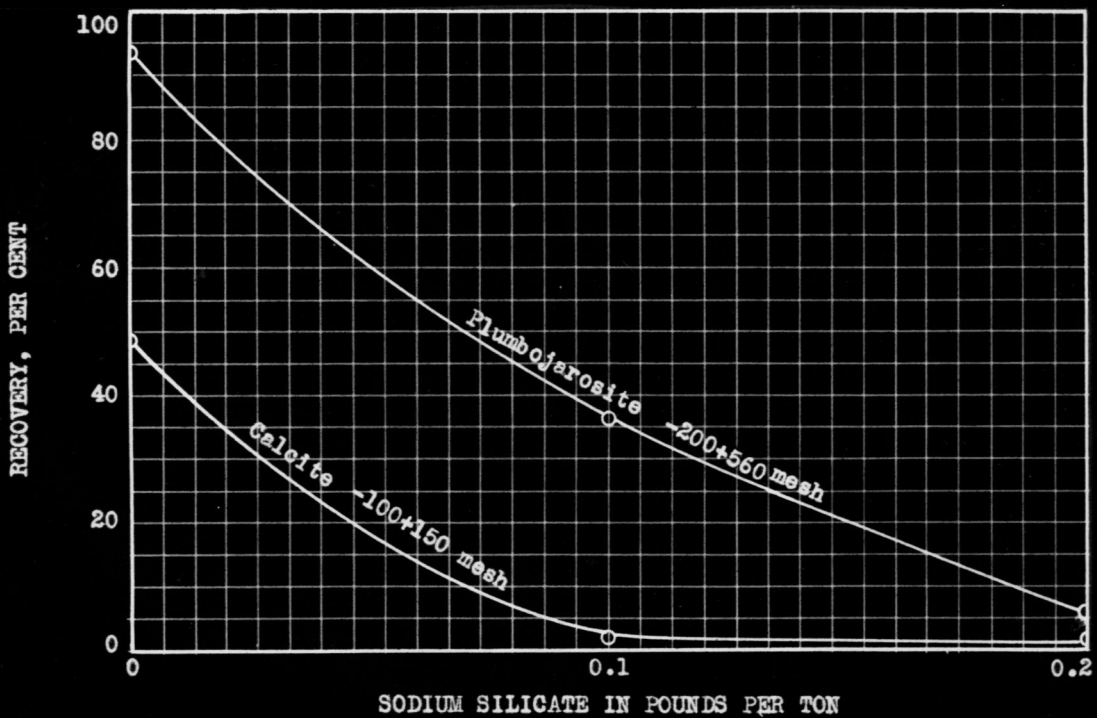

Fig. 15. Effect of sodium silicate on the differential flotation of synthetic 1 to 1 mixtures of calcite and plumbojarosite using 0.075 pound oleic acid and 0.5 pound terpineol per ton of ore.

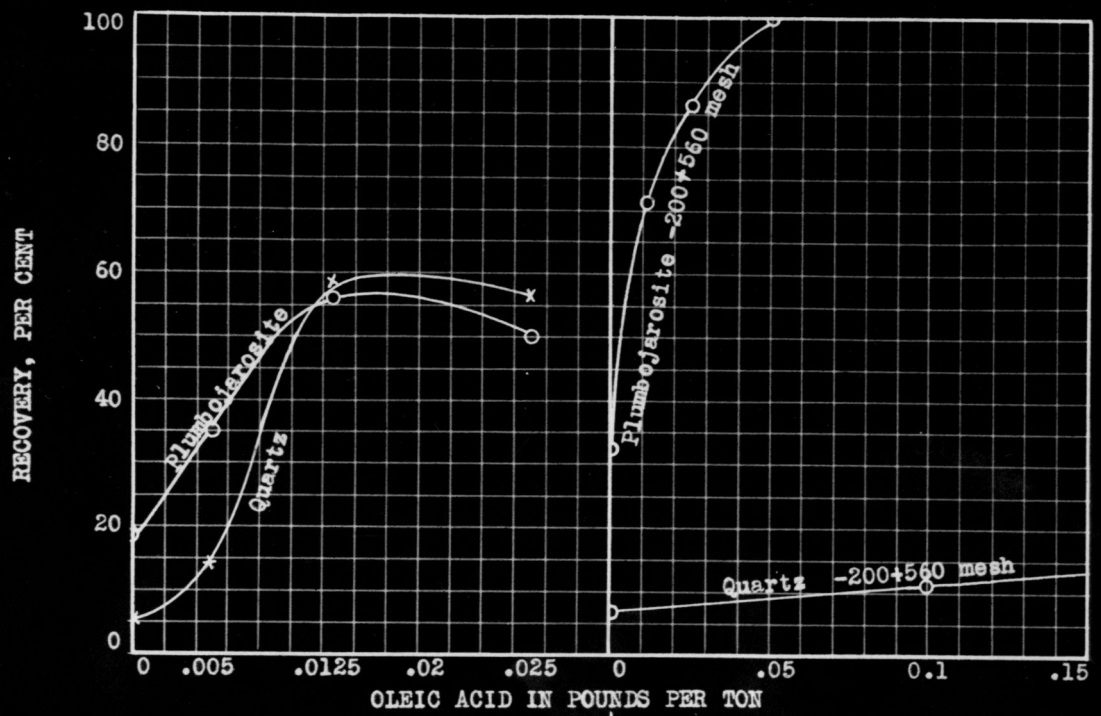

Fig. 16. Effect of oleic acid on the differential flotation of synthetic 1 to 1 mixtures of quartz and plumbojarosite with 0.5 pound terpineol per ton of ore.

Fig. 17. Flotation of pure plumbojarosite compared with flotation of pure quartz using various concentrations of oleic acid with 0.5 pound terpineol per ton ore.

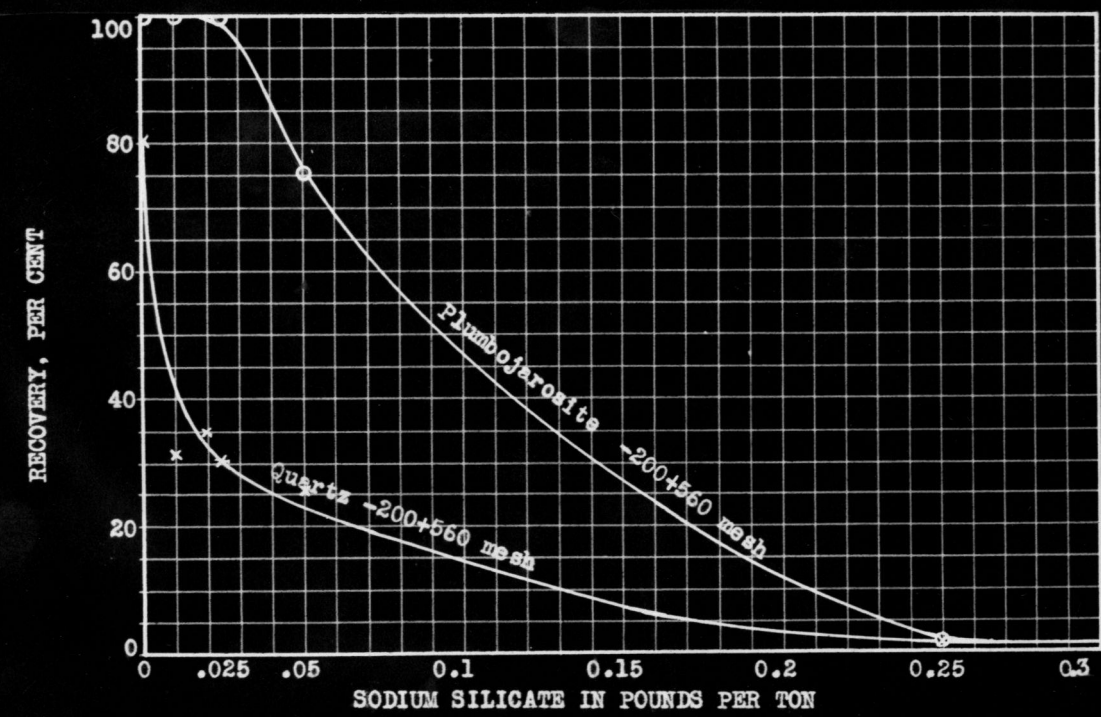

Fig. 18. Effect of sodium silicate on the differential flotation of synthetic 1 to 1 mixtures of quartz and plumbojarosite using 0.025 pound oleic acid and 0.5 pound terpineol per ton of ore.

XANTHATES USED

Name	Formula	No. carbon atoms
n-K-ethyl	$CH_3CH_2-O-C(=S)-SK$	2
n-K-amyl	$C_5H_{11}-O-C(=S)-SK$	5
n-K-octyl	$CH_3(CH_2)_7-O-C(=S)-SK$	8
n-K-lauryl	$CH_3(CH_2)_{11}-O-C(=S)-SK$	12

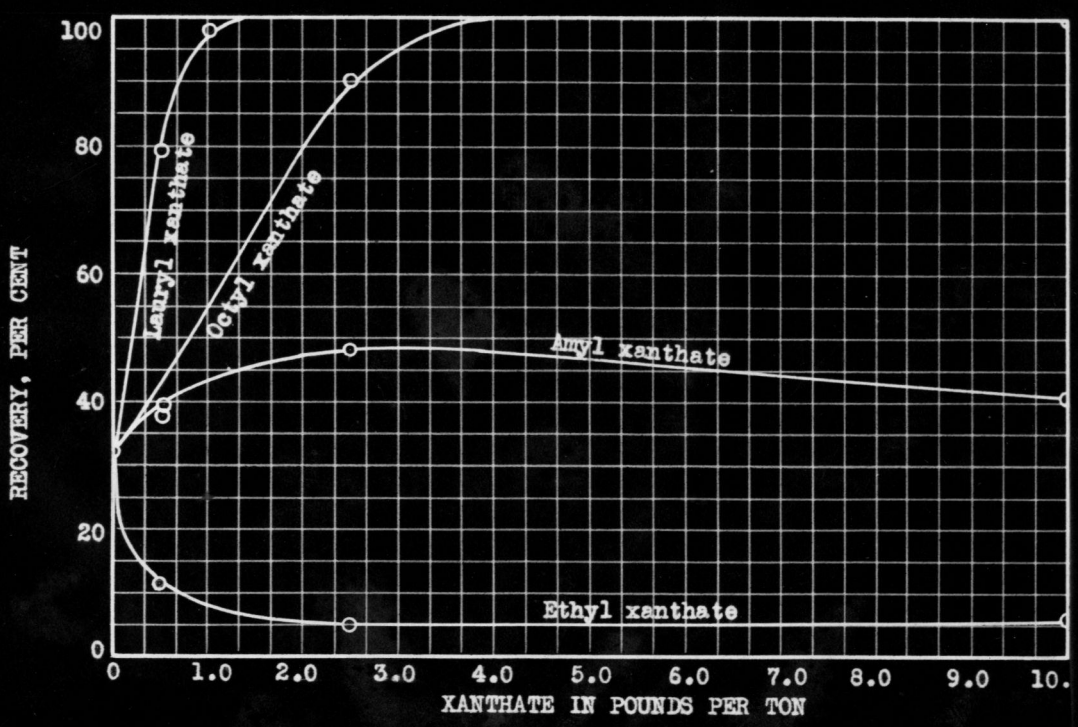

Fig. 19. Effect of various concentrations of some of the xanthates on the flotation of minus 200 plus 560 mesh plumbojarosite with 0.5 pound terpineol per ton of ore.